Westinghouse Research Laboratories
Pittsburgh, Pennsylvania 15235

FURNACE AND SUPPORT EQUIPMENT FOR SPACE PROCESSING

R. Mazelsky, C. S. Duncan, R. G. Seidensticker,
R. A. Johnson, R. H. Hopkins and G.W. Roland

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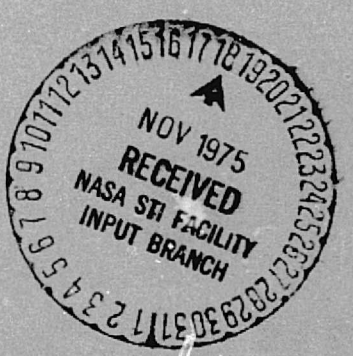

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1. INTRODUCTION

Materials processing in space is a relatively new concept proposed to apply the unique environment available in orbiting space craft toward the development of new materials or processes of commercial applicability. The most interesting parameter for exploitation is the effect of free fall conditions in eliminating density-induced convection in fluids or gases and permitting surface tension forces to dominate in unconstrained fluids. Just the consideration of these two effects have a profound impact on the physical characteristics, solidification and nucleation modes, and processing mechanisms of materials. This has already been verified by the opportunities presented for materials studies on the Apollo and Skylab missions.

There have been considerable constraints placed on materials studies because of the restricted facilities available for equipment imposed by limitations of weight, power, volume, and time. As a result, the scope of materials processing investigations has been confined to limited numbers of experiments on materials within limited temperature ranges. The Shuttle missions beginning in the 1980's present an opportunity of expanding the scope of materials studies and providing baseline information on chemical systems, processing parameters, and equipment requirements necessary for manufacturing processes in space.

2. STUDY OBJECTIVES

Although the prime thrust of the program is to give clearer definition to the furnace subelement for Space Processing Applications, the end product is to provide a facility for doing materials research, product development, and ultimately processing and manufacture. There is essentially an infinite combination of equipment-material system combinations that can be conceived. Our objective is not to list these options but basically to define a core facility capable of performing a majority of materials processing functions. This definition is oriented toward a laboratory-type facility for the purpose of obtaining information and defining process parameters leaving pilot plant and preproduction processes be its own definition for required equipment. Our object, then, is to categorize the experiment classes, describe the needs peculiar to each experiment type and to project facility requirements to perform the experiments. As constraints, we have adopted sample sizes large enough to provide meaningful data, existing technology as an equipment base, and efficiency with regard to weight, volume, and power.

3. RELATIONSHIP TO OTHER NASA EFFORTS

As an information base, we have used the reports from NAS 8-28938 and NAS 8-30741. Information derived from Skylab and ASTP missions yielded a definition of the experimentors' requirements for furnace performance and data collection. MSFC reports on furnace development were used when available in order that current thinking be incorporated.

4. METHOD OF APPROACH

Since the actual materials experiment is the end product of the furnace subelement activity, we have elected to consider equipment based on a definition of materials processes. The processes were developed as a matrix with parameter ranges required to satisfactorily perform the process. Equipment selection was then determined by what must be available to meet the requirements of the matrix. Typical equipment available within present technology was selected and equipment performance evaluated. Finally, the compatibility of the different materials processes with equipment parameters and the free fall environment was evaluated and typical experiments were examined.

5. DATA GENERATED AND SPECIFIC RESULTS

5.1 Materials Processes

The matrix of materials process versus operating parameters developed earlier is reproduced here. We have attempted to bracket those parameters that are in a probable category so as not to attempt to define all potential experiments. Based on this matrix we can project specific types of experiments and the requirements to perform them.

5.1.1 Melt Cast

It would be safe to assume that casting experiments done during early missions would not be oriented toward obtaining specific shapes but would be oriented more toward obtaining a specific grain structure or texture that would later be processed in the solid. The critical points in the process would be:

1. Heating in position to assure complete melting and homogeneity.
2. Translation of molten material into a mold.
3. Retaining contact of the fluid with the mold.
4. Providing the desired direction and rate of heat extraction.

The melting process is easily accomplished by furnaces, electromagnetic heating, electron beam heating or imaging. The translation can be done by positioning devices either mechanical or electromagnetic. Maintaining contact with the mold would require application of force until solidification is complete. Heat extraction is accomplished by specific mold design.

There are several possible alternatives for performing the above functions, all of which require at least some operator participation. A possible concept that could be automated may be the use of a

TABLE 1

		Melt-Cast	Bridgeman	Gradient Freeze	Isothermal	Czochralski	Float Zone
Maximum Temperature Range	Metals 3700 (2000)	—	—	—	—	—	—
	Glasses 2800 (1800)	—	—	—	—	—	—
	Ceramics 3500 (2300)	—	—	—	—	—	—
	Semicond 2500 (1500)	—	—	—	—	—	—
Growth Parameter	Gradient °C/cm	X	5-300 (10-70)	5-100 (10-70)	Sol'n-imposed grad	10-150 (30-100)	30-200
	Growth Rate cm/hr	X	.03-100 (.1-30)	.03-100 (.1-30)	< 0.01 cm/hr	.025-40 (.2-15)	.3-40
	Rotation rpm	X	X	X	0-1000	10-300 (25-100)	10-100
Atmosphere, Pressure	Vacuum Oxidize Neutral Reduce	Chemical System Defined	—	—	—	—	—
Temperature	Control °C	± 10	~ ± 1	~ ± 1	Solution growth ± 0.5	< ± 1	< ± 1
Temperature Measurement	Deadband °C	± 1	—	—	—	—	—
	Calibration %	± 0.75	—	—	—	—	—
Manipulation		positioning fluid containment sample motion	positioning furnace motion sample motion	positioning	positioning Sol'n growth-seed motion	seed positioning charge positioning seed motion crucible rotation	sample positioning sample motion sample rotation
Programming	Heat Up	—	—	—	—	—	—
	Soak	—	—	—	—	—	—
	Cool	—	—	—	—	—	—
	Cycle	X	X	X	X	X	—
Data Access and Record	Temperature	—	—	—	—	—	—
	Time	—	—	—	—	—	—
	Position	X	—	—	solution growth —	—	—
	Atmosphere as required	—	—	—	—	—	—

piston-like heating chamber shown on Fig. 1. The sample after melting is maintained at temperature for homogenization. The piston is within a heat leveler to maintain a reasonably small temperature gradient. For casting the piston is compressed and gas pockets are removed by a pressure release. The furnace is shut-off and cooling gas or fluid is injected at the mold end. Concurrently, cooling gas is inserted into the furnace if desired.

In order to retain the versatility of the furnace, it would probably be desirable to design the casting assembly so as to be integral with a set of furnace base plates. This would require coupling and decoupling mechanical and gas or liquid fittings which would add to automation functions. From that point on, the process can be programmed. One obvious difficulty with this concept is materials compatibility. There will inevitably be forces which will cause sample motion resulting in cylinder wall contact. As a result, a non-wetting material for the assembly is a requirement. Another limitation is the fact that cooling is performed within a furnace. As a result, cooling rates will be limited by the thermal capacity of the system. In situ freezing would require a less complex assembly but would require a modification of the cooling mechanism.

5.1.1.1 Melt Casting Experiment

Run Procedure:

1. Remove specimen assembly from stowage.
2. Install specimen assembly in furnace chamber.
3. Evacuate furnace chamber; repressurize with desired ambient; (oxidizing, reducing or inert gases).
4. Set furnace heat up and soak temperatures; times.
5. Initiate melt down and soak cycles (This step may be preceded by electromagnetic levitation-manipulation of the sample if containerless melting is employed.
6. Soak sample at temperature for specified time.

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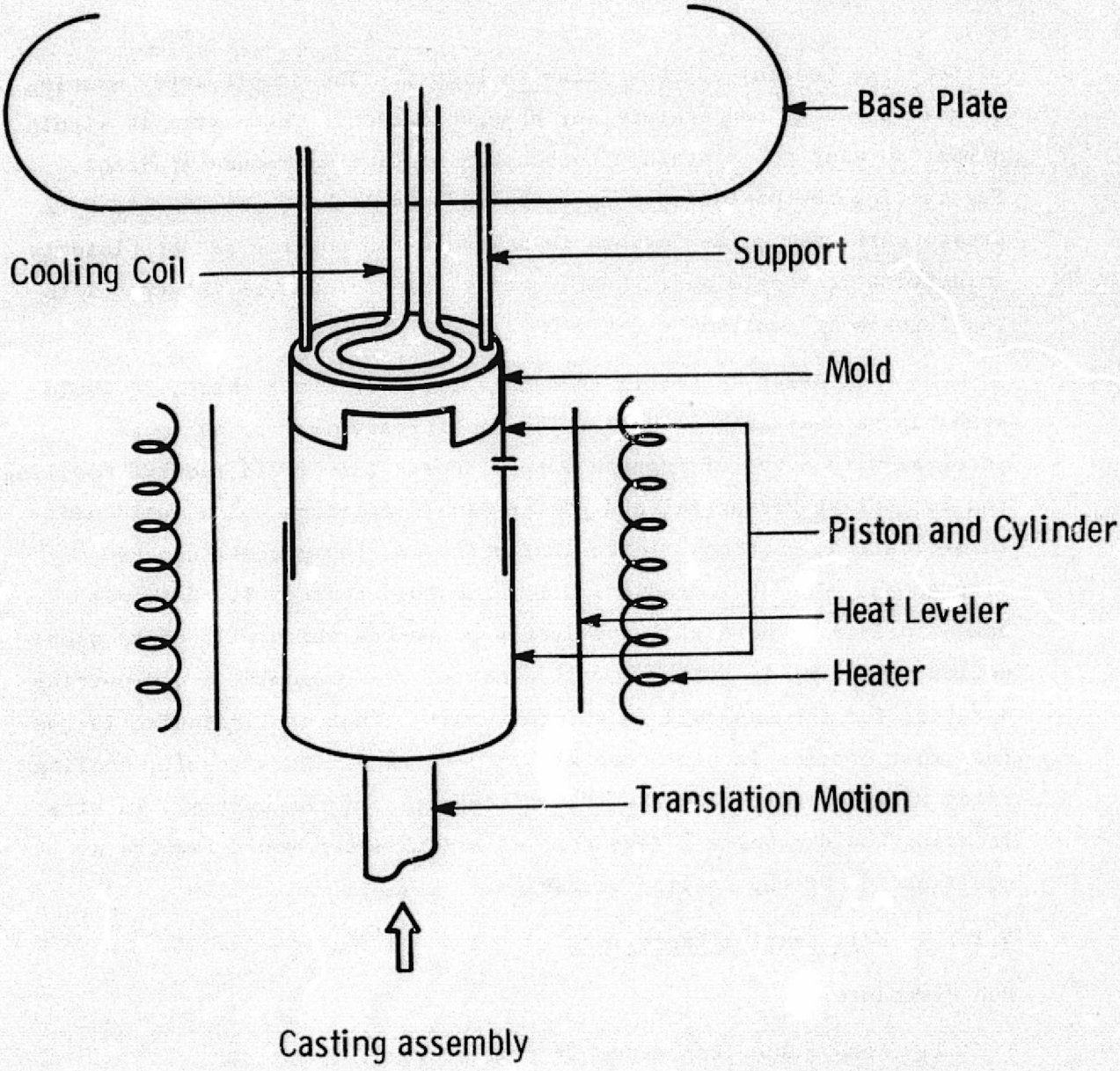

Figure 1

*7. Freeze sample In Situ or activate piston motion prior to freezing.

8. Activate mold cooling.

9. Cool apparatus to touch temperature.

10. Repressurize system to ambient.

11. Remove specimen assembly.

12. Stow specimen.

5.1.1.2 Examples

A. Fe-Ni-Cr M.P ∿ 1450; superheat 150°C.

B. Soda lime glass (∿ 75% SiO_2, 15% Na_2O, 12% CaO) M.P ∿ 1000°C; superheat 100-200°C.

Long soak periods were chosen to assure homogenization of specimens. This time will be a function of specimen preparation and composition.

5.1.2 Bridgman Growth

This growth technique is in essence similar to that performed in normal gravity in that the material is contained, usually in a cylindrical configuration. The basic process involves the translation of the molten sample through a hot zone maintained above the melting point of the material into a cooler zone below the melting point but usually above room temperature. The passage through the temperature gradient results in a directional solidification of the material.

The important process parameters are:

1. Sample translation is accomplished with low vibration at a controlled rate in order to achieve a near linear solidification velocity.

2. The temperature gradient is controllable so as to impose on the sample a gradient satisfactory to satisfy the experiment goals. For some materials gradient requirements of 100-150°C/cm are not unusual.

3. The heat flow patterns should be controllable in order to maintain the desirable solid-liquid interface shape.

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*This step may be achieved by quenching or slow cooling to anneal the sample.

The temperature requirements can be met by the use of a multiple zone furnace or a single zone furnace with the solidification occurring in the temperature gradient imposed by end losses. Care should be taken to assure that the support rod attached to the sample container to provide translation is not a perturbing influence on heat flow. Precise positioning of the charge is critical only insofar as a specific molten length is required.

5.1.2.1 Bridgman Experiment

Run Procedure:

1. Remove experiment from stowage.
2. Attach to support and translation assembly.
3. Insert experiment in furnace.
4. Furnace power on for both temperature zones.
5. Record and verify temperature profile.
6. Activate translation mechanism for predetermined growth rate.
7. When entire sample is in Zone 2, deactivate translational mechanism.
8. After predetermined anneal time, shut off or program down furnace power.
9. Remove experiment assembly from furnace.
10. Detach sample and stow.

5.1.2.2 Examples

A. Sulfosalts, i.e., Ag_3AsS_3, Tl_3AsSe_3; melting points $\sim$ 400°C.
B. Alkali Halides, i.e., NaCl, KCl; melting points $\sim$ 900°C.
C. Metal Alloys and Eutectics, i.e., Ni, Cr; melting points $\sim$ 1400°C.

5.1.3 Gradient Freeze

Like the Bridgman process, the gradient freeze has essentially the same parameters and configuration that would be used on earth. It is simpler for automated processing than other techniques in that no rotational or translation motion is required. It is most appropriately

done in a container and is, therefore, limited as is the Bridgman process by the existence of compatible container materials; i.e., non-wetting, non-corrosive and non-contaminating interfaces with the sample material. The process involves the imposition of a thermal gradient along a sample such that a portion or all of the sample is melted. The temperature along the gradient is then programmed downward resulting in a translation of the melting temperature along the sample. As a result, the material is directionally solidified.

Critical parameters that must be controlled are:

1. Heat flow into and out of the sample must vary monotonically in order to maintain a nearly linear interface velocity.
2. Heat flow should be predominantly in an axial direction in order to maintain a nearly planar solid-liquid interface.
3. Temperature control must be stable (without major oscillation) to assure no variation at the solid-liquid interface.

Two types of equipment are most appropriate for doing this type of process. One is the multizone type furnace where each zone is independently controlled and programmed. The other is the MA-010 type furnace where the program cooling is a time varying function thereby correcting changes in the interface velocity inherent in solid-liquid systems having single point control. Since the sample itself is not in motion, this process lends itself to multipoint temperature sensing, temperature shaping accessories, reproducible positioning, and automated feedback and temperature control.

Although not strictly a gradient freeze diffusion along a temperature gradient is a similar process having similar configuration. Processes in this category are simple diffusion along a gradient and solute transport along the gradient and subsequent crystal growth. The latter process depends on having a differential solubility at different temperature permitting dissolution of polycrystalline material at the hot end and controlled exsolution at a colder portion. In effect, the

temperature gradient sets up a concentration gradient that should permit steady state growth on a seed or self-nucleated crystals.

Another form of gradient crystal growth similar to solution growth is vapor transport. Once again the driving force is the temperature gradient which causes mass transport of volatile species from the hot to the cold end. If a transport agent such as iodine is used, the gaseous iodides react in the cooler portion by dissolution and compound formation releasing the carrier or iodine. This migrates to the source or hot end causing further transport.

In both the above examples the temperature gradient is kept stable and is not a time varying function. Both rely on a steady state situation whereby the reactive mass arrives at the growing interface at a rate slow enough to be accommodated in a crystalline array. The primary driver is that source material and the growing crystal be maintained at temperatures commensurate with mass transport and reaction onto a crystalline structure.

5.1.3.1 Run Procedure

1. Remove specimen assembly from stowage.
2. Insert specimen assembly in furnace cavity.
3. Obtain required atmosphere, e.g.,
   a) evaluate system
   b) flush with desired gas ambient*
   c) set gas flow for run .
4. Set soak time and temperature; set programmed cooldown cycle. Programmed cool cycle is not required in steady state gradient growth process such as solution growth and vapor transport.
5. Initiate heat up, soak and programmed growth cycles.
6. Terminate run, initiate cooldown cycle.

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*inert (argon, nitrogen), reducing (H_2), or oxidizing (oxygen, oxygen/nitrogen mixtures) or lab atm if sample in sealed container.

7. When cool, adjust furnace ambient back to that of lab environment.
8. Remove ingot-crucible assembly and stow.

5.1.3.2 Examples

A. Eutectic Composites, e.g. Fused salt.
M.P ∿ 1000°C, gradient ∿ 50°C/cm.

B. Semiconductors, e.g. Silicon
M.P ∿ 1400°C, gradient ∿ 50°C/cm.

C. Sulfosalts, e.g. Proustite
M.P ∿ 350°C, gradient 120°C/cm.

D. Solution Growth, e.g. GaAs-Ga
Temperature ∿ 900°C, gradient ∿ 30°C/cm.

E. Vapor Transport, e.g. GeSe(I_2)
Temperature ∿ 700°C, gradient ∿ 20°C/cm.

5.1.4 Isothermal Processing

A broad range of different types of material objectives can be included in this category. In the broadest terms, this process entails the melting of a material and maintenance at temperature for some length of time in an essentially isothermal environment. For example, casting in which the melt is not solidified in a mold is in this category. The main distinguishing factor is that most processes in this category would require a slow cool as opposed to quenching.

The furnace equipment required to do the melting is not specialized and what is used is primarily a function of the temperature needed. However ens'tive temperature programming may be required. For example, glass ormat'on and alloy systems are typical in that the material is melted and maintained at temperature to promote homogenization. In the case of glasses or other potentially low thermal conductivity materials cooling rate is limited by the heat flow out of the solidified melt in that too large a thermal gradient from the center to the edge may cause strain and cracking. In higher thermal conductivity systems cooling rates may be governed by a specific phenomenon such as controlled precipitation or solidus exsolution during cooling.

A special case on this category is solution growth which is essentially an isothermal process with a temperature gradient superimposed at the desired nucleation site. A classical system is the techniques used to grow magnetic garnet crystals such as YIG. A complex solvent comprised of $PbO-PbF_2-B_2O_3$ forms a solution with $Y_2O_3-Fe_2O_3$ in a platinum crucible. A "cold" spot is generated by a gas flow in an otherwise isothermal system. This specifies a nucleation area and minimizes surface seeding and growth. The temperature is programmed downward to exceed the solubility limit and promote growth preferentially in the cooler area. At the completion of the programmed cool, a plug is removed and the liquid is drained. This is to prevent strain in the crystals caused by solidification of the solvent when the assembly is cooled further. $BaTiO_3$ growth in KF frequently involves pouring the molten solvent off after crystal growth but before cooling to room temperature. Other modifications involve dipping a seed in the solution and propagating growth on a seed crystal with subsequent removal of the crystal from the solution prior to solution solidification. A specific application of this technique is "top-seeded" liquid phase epitaxy.

Performance of these experiments require sophisticated program temperature control and a mechanism for separation of the liquid from the solid while hot. Since there is no driving force for this separation in free fall, the experiment assembly must provide one. One might consider some concepts to accomplish this.

One driving mechanism is centrifugal force. This would require a rotation mechanism either to spin the whole assembly and permit the liquid solution to be expelled through orifices or withdraw the crystal and spin it to remove liquid. Either mechanism requires substantial rotation speeds of several hundred revolutions/minute, a mechanical as well as a vibrational problem.

Another alternative is to use a vacuum to withdraw the fluid. This would require a shutter type arrangement in a line leading from the

crucible. It would be kept closed during growth and then opened to permit a vacuum.

A third option is to use a bundle of capillaries made of material wet by the solvent.* At the completion of growth the capillaries would be inserted in the melt which would be withdrawn by capillary action.

All of the alternatives presented require some translational or rotational motion within a potentially corrosive system which may require hermeticity. As a result, such concepts can be utilized only when chemically compatible materials are available at the temperature range of interest.

5.1.4.1 Run Procedure

Isothermal Melting:

1. Remove experiment from stowage.
2. Insert in furnace.
3. Furnace-control system activated.
4. Control cool activated.
5. Control deactivated-furnace cool.
6. Remove sample.
7. Stow.

Solution Growth:

1. Remove experiment from stowage.
2. Insert experiment in furnace.
3. Furnace-control system activated.
4. Soak 24 hours.
5. Activate gas flow to cold-finger seed (if desired).
6. Activate slow-cool program
7. Interrupt cooldown.
8. Remove solvent (if required).
9. Initiate new cooldown to desired temperature.

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*Communication -- R. Hammel, TRW.

10. Shut off power.
11. Remove experiment from furnace after temperature reaches ambient.

LPE Growth:

1. Remove experiment from stowage.
2. Insert experiment in furnace.
3. Furnace power on to soak-power level.
4. Measure soak temperature after predetermined time interval.
5. After 12 hours, reduce power to growth-power level.
6. Measure growth temperature after predetermined time.
7. Mate substrate with liquid phase.
8. After desired growth period, activate mechanism to separate substrate and liquid phase.
9. Rotate substrate to remove fluid droplets.
10. Shut off furnace power.
11. Remove experiment from furnace after temperature reaches ambient.

5.1.4.2 Examples

A. Solution Flux Growth, e.g. YIG
   Temperature 1200-1300°C ∿ 900°C.
B. Liquid Phase Epitaxial Growth, e.g. Magnetic Garnet
   Temperature 1300°C ∿ 950°C.
C. Isothermal Melting, e.g. Si-Ge
   Temperature 1000-1500°C.

5.1.5 Czochralski Crystal Growing

The Czochralski method for crystal growing is the most widely used for both laboratory and commercial purposes. In its broadest concept, the Czochralski method may be defined as unconstrained growth of a crystal by means of its controlled withdrawal from the melt. In the conventional one-gravity application, gravity is utilized to keep

molten raw material contained within a crucible. Because gravity is not in effect in space flight, unique variations of the Czochralski process must be devised in order to allow its use.

Several variations of the Czochralski method are suitable for space flight application and, for descriptive purposes, may be classified by the following types:

- Crucible
- Liquid Cavity (or Self-Crucible)
- Molten Zone.

Crucible Type

In appearance, this variation of the Czochralski method most nearly resembles the more familiar earth-based applications. A crucible may be used at zero-gravity, but only if a mechanism is provided which will confine the melt within the intended portion of the crucible. In order that this may be done, two conditions must be satisfied. First, the intended portion of the crucible cavity must have the property of wetting the molten material from which the crystal is to be grown. This condition is necessary because it provides a force which adheres the liquid to the crucible. In zero-gravity, this effect in combination with surface tension will keep the material situated in the crucible. The second requirement is the placement of a non-wetting barrier which prevents flow of the melt over the entire (inside and outside) crucible surface. When these conditions have been satisfied, crystals may be grown by apparatus and techniques fundamentally similar to those used at one gravity. This variation of the Czochralski process is depicted in Fig. 2.

This variation of the Czochralski method suffers from two major disadvantages. First, fully automated control would be very difficult to provide and, secondly, acceptable crucible materials probably do not exist for a great many of the desirable crystal materials. The principal advantage of this method, when applicable, is that it will allow otherwise identical growth at one-gravity and in space.

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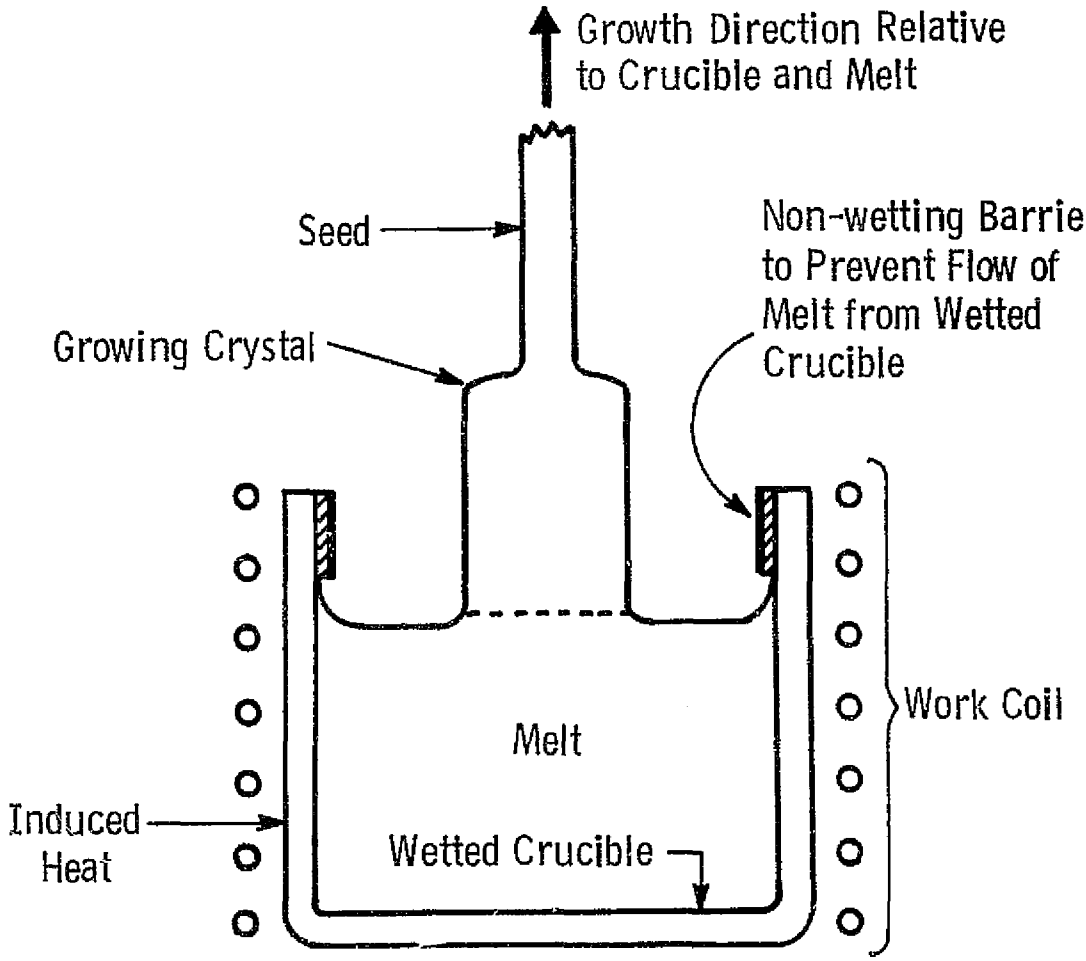

Schematic representation of crucible type Czochralski crystal growth method as adapted for space flight (Induction heating shown for purpose of illustration. Other methods of heating also acceptable)

Figure 2

Liquid Cavity

Crystal growth by this scheme has been performed, although only rarely, at one-gravity and is quite limited in its application. The basic requirement of this method is to create a molten cavity in a relatively large solid mass of the raw material from which a crystal is to be grown. Surface tension and adherence to the solid mass will, in zero gravity, hold the liquid in place. After the liquid cavity has been formed, crystal growth may be performed in a manner similar to that of earth-based Czochralski processes. Heat to form the cavity must be supplied by some form of directed or focussed energy such as by optical or electron beam. See Fig. 3.

This variation of the Czochralski method also has the disadvantage of limited application because of several inherent problems. The method is relatively wasteful of material because only a small portion of the raw material can be converted into crystal and crystals grown by this method will tend to be small as compared to other methods. Also, large amounts of properly focussed energy as required for this mode of growth are relatively expensive. Finally, the method is difficult to automate. This method can be duplicated exactly in one gravity and space.

Molten Zone

Growth by this method at zero gravity is, in appearance, much like floating zone growth performed at one-gravity. The absence of gravity, however, has a profoundly favorable effect upon the stability of the molten zone. In one-gravity floating zone crystal growth a minimum of three important forces act upon the molten zone; two of these forces have a stabilizing effect and one has an unstabilizing effect. The two stabilizirg forces are: a) surface tension, which holds the zone intact, and b) adherence by wetting, which holds the zone in place and in contact with the growing crystal and the raw material feed rod. Gravity is an unstabilizing force which acts to pull the zone apart and away from its intended position. If induction heating is used, an

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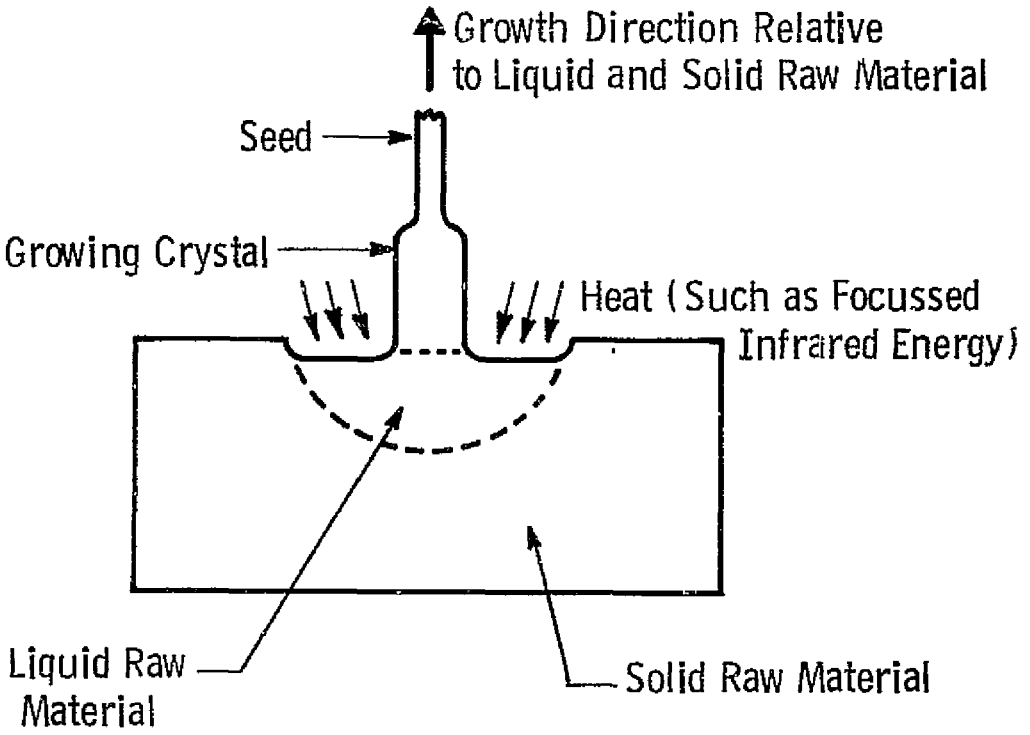


Schematic representation of liquid cavity variation of Czochralski method as adapted for space flight

Figure 3

additional stabilizing force, the resultant field effect, may act upon the zone. The molten zone variations of the basic Czochralski process are shown schematically in Figs. 4a, 4b, and 4c.

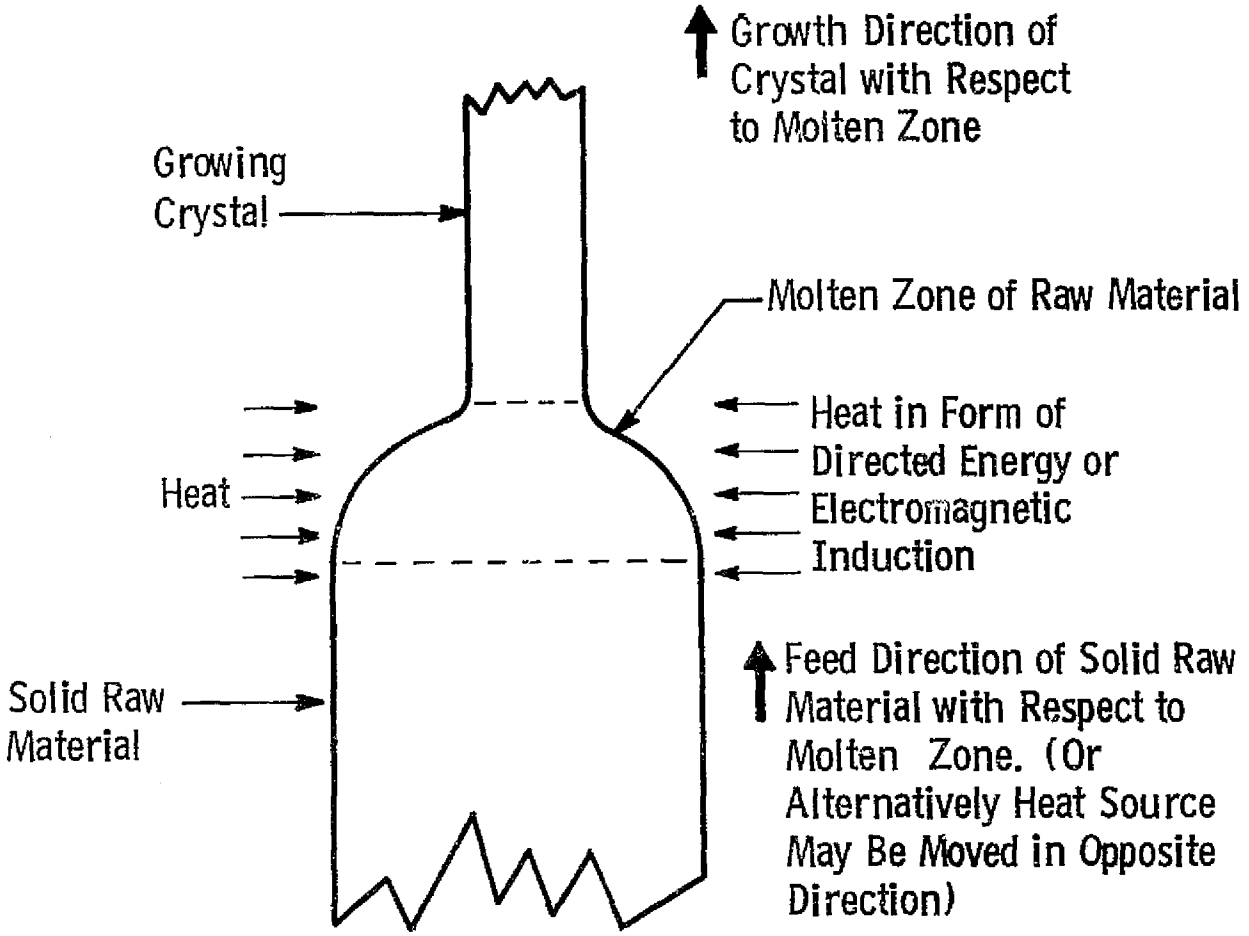
Figure 4a depicts the molten zone application which most nearly resembles a conventional Czochralski growth. In this instance the molten end of the feed rod serves, similar in purpose to that of a crucible, as a reservoir from which a growing crystal is withdrawn. Figure 4b illustrates a reverse ratio of diameters with the molten zone stability being largely provided by the growing crystal. In each of these cases it is necessary, during growth, to provide for different rates of travel into and out of the zone by the raw material feed rod and the growing crystal. Figure 4c shows a common diameter molten zone arrangement which is similar to one-gravity floating zone systems, such as those used for silicon. The common diameter system functions with synchronized movement of the feed and growth rods with respect to the molten zone.

Because of the absence of the force of gravity on the molten zone, this method offers many space flight experiment opportunities. Any material which can be grown by floating zone in one-gravity can be grown as well or better in zero gravity. More importantly, a large number of materials which cannot be grown by floating zone in one-gravity because of their medium to high specific gravity, on the contrary, offer little or no difficulty at zero gravity.

Heat required for this process can be supplied by either high frequency induction or some form of focussed energy such as by optical or electron beam methods.

As with all floating zone techniques, an important advantage of this method is the absence of crucibles and the attendant problems.

Apparatus for space flight applications of this process can be designed basically similar to the one-gravity systems, in particular the systems which are used for growing silicon. The common diameter



Schematic representation of molten zone variation of Czochralski method as adapted for space flight-diameter reducing version

Figure 4a

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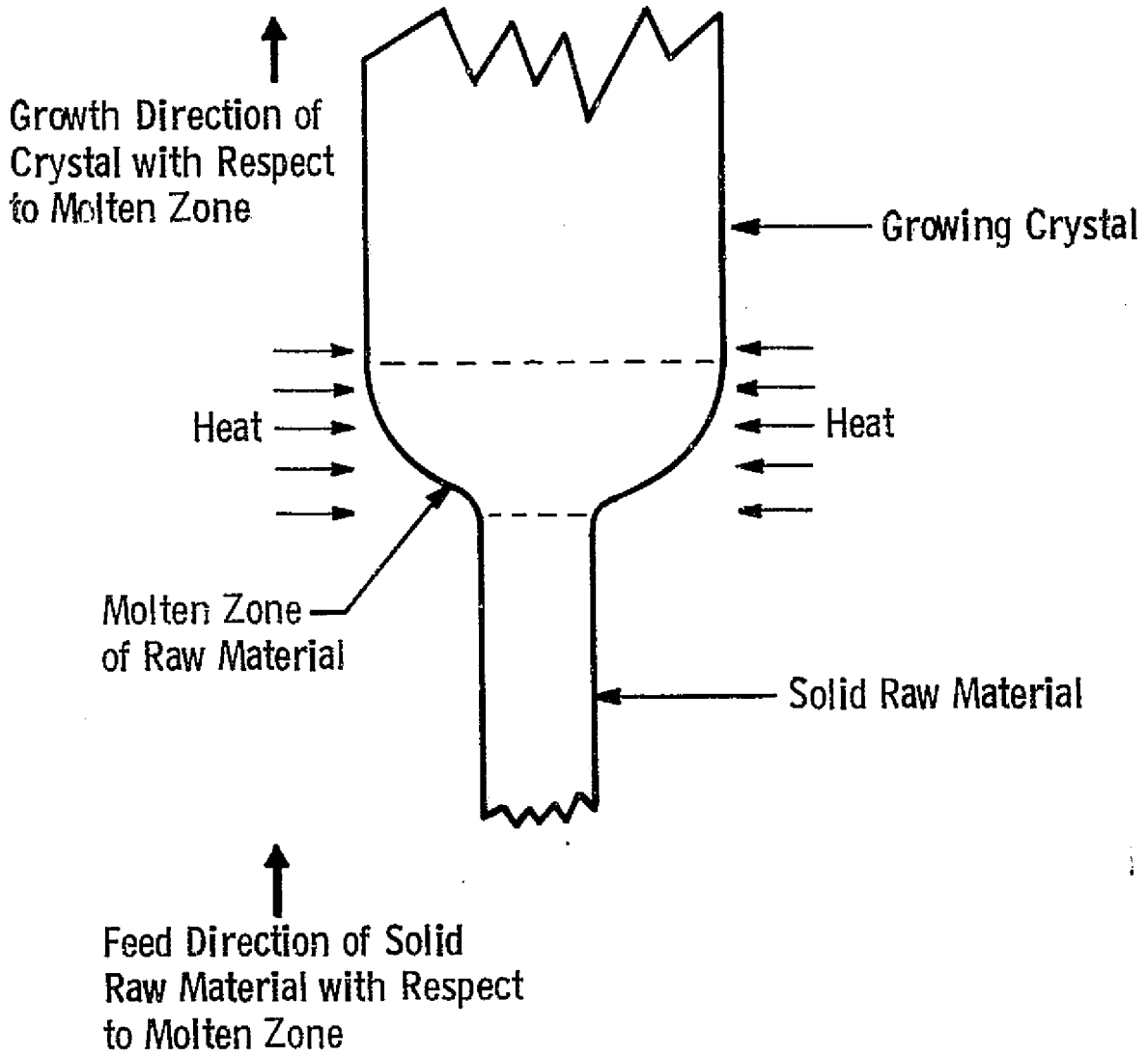

Schematic representation of molten zone variation of Czochralski method as adapted for space flight - diameter increasing version

Figure 4b

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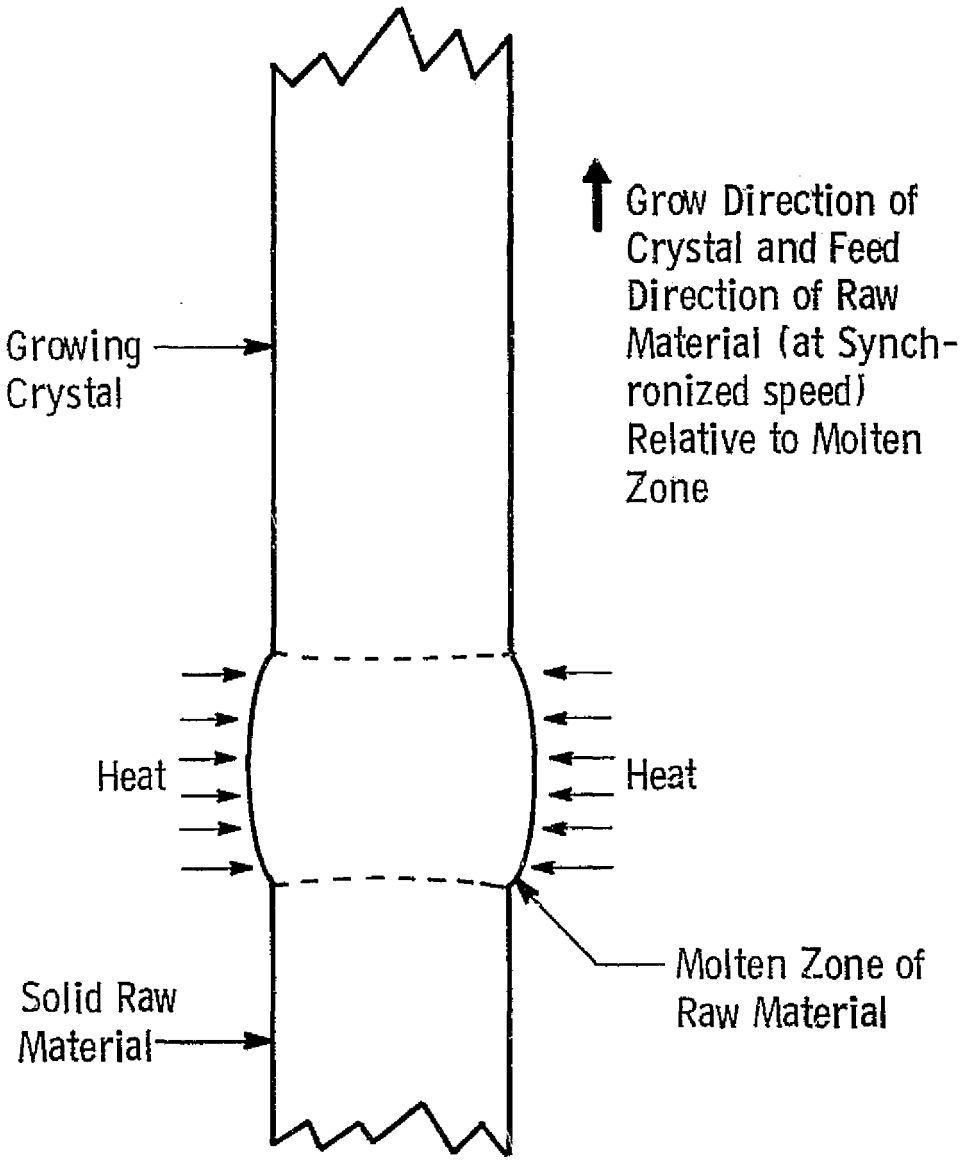

Schematic representation of molten zone variation of Czochralski method as adapted for space flight - common diameter version

Figure 4c

version of this method, Fig. 4c, is somewhat simpler because a common growth and feed rate is required. The common diameter version also has the advantage of simpler heating control requirements and in some instances is readily adaptable to fully automated, unmanned control of the entire growth cycle.

Of the Czochralski methods which are acceptable for space flight use, the molten zone types are the best suited for adaptation to automatic control. Among the molten zone types, the common diameter version is by far the most easily automated. The common diameter version can, in selected applications, be automated totally on the basis of earth-based characterization. This is possible because the entire growth can be performed by programming a series of fixed conditions which do not require the use of closed loop control systems. For example, a predetermined program can, in chronological order, (1) establish atmospheric ambient, (2) apply a predetermined level of heater power at the growing crystal/raw material interface, (3) hold for a predetermined period of time to allow the molten zone to form and equilibrate, (4) apply, for a predetermined rate and period of time, axial motion to the zone with respect to the growing crystal and the raw material feed rod, (5) deenergize heater (preferably slowly), and (6) deactivate atmospheric ambient. For more difficult applications such, for example, as marginally large diameter, different growth and raw material feed diameters, or multiple zone passes, more complex closed loop control systems and programs would be required.

Comparatively complex automatic program systems as, for instance, a diameter reduction system, Fig. 4a, must provide for needs which do not occur with the simpler common diameter system discussed in the previous paragraph. Unlike the essentially equal induction heating which occurs when common diameters are used, the heating of unlike diameters differs greatly and, as a consequence, the small rod, because of its relatively poor coupling to the induction field, may never reach its melting point or form a mutual molten zone with the larger raw

material feed rod. Usually in earth-based laboratories this difficulty is surmounted by careful operator control of work-coil positioning and heating power level adjustment until a connected zone is formed. For unmanned space flight application, a predetermined heating power level suitable for growth is also suitable for forming the starting molten zone provided a concentric joint is prepared at the seed/feed rod interface. See Fig. 4d. As it is important to maintain an essentially constant molten zone volume, it will be necessary to drive the growing crystal and raw material rods at rates inversely proportional to their cross-sectional areas. It will likely be necessary to have a closed-loop diameter control system to determine the travel rates, as needed, to assure a constant growth diameter. The sensing transducer for this purpose may be an infrared detector utilized somewhat as with earth-based applications of Czochralski growth. The detector would be focussed at the growing liquid/solid interface and would remain fixed with respect to the molten zone position.

In general, the mechanical hardware requirements of the molten zone method in space flight are quite similar to floating zone methods used at one gravity. The material processing portion of the system must have provision to apply the required ambient atmosphere, including vacuum, as dictated by the needs of the crystal growth material. The raw material and the growing crystal mounting fixtures must, except in special cases, be capable of rotation, when required, either synchronously or independently, including reverse direction. Refer to Fig. 4d. The linear motion of the material mounting fixtures must be capable of synchronized motion as well as predetermined differential rates of motion whenever different material diameters are used. If desired, motion of the heating and molten zone may be used, especially with the common diameter method, as a means for obtaining all or a portion of the relative motion needed.

Heating may be supplied by high frequency electromagnetic induction or by properly directed electron beam or optically focussed

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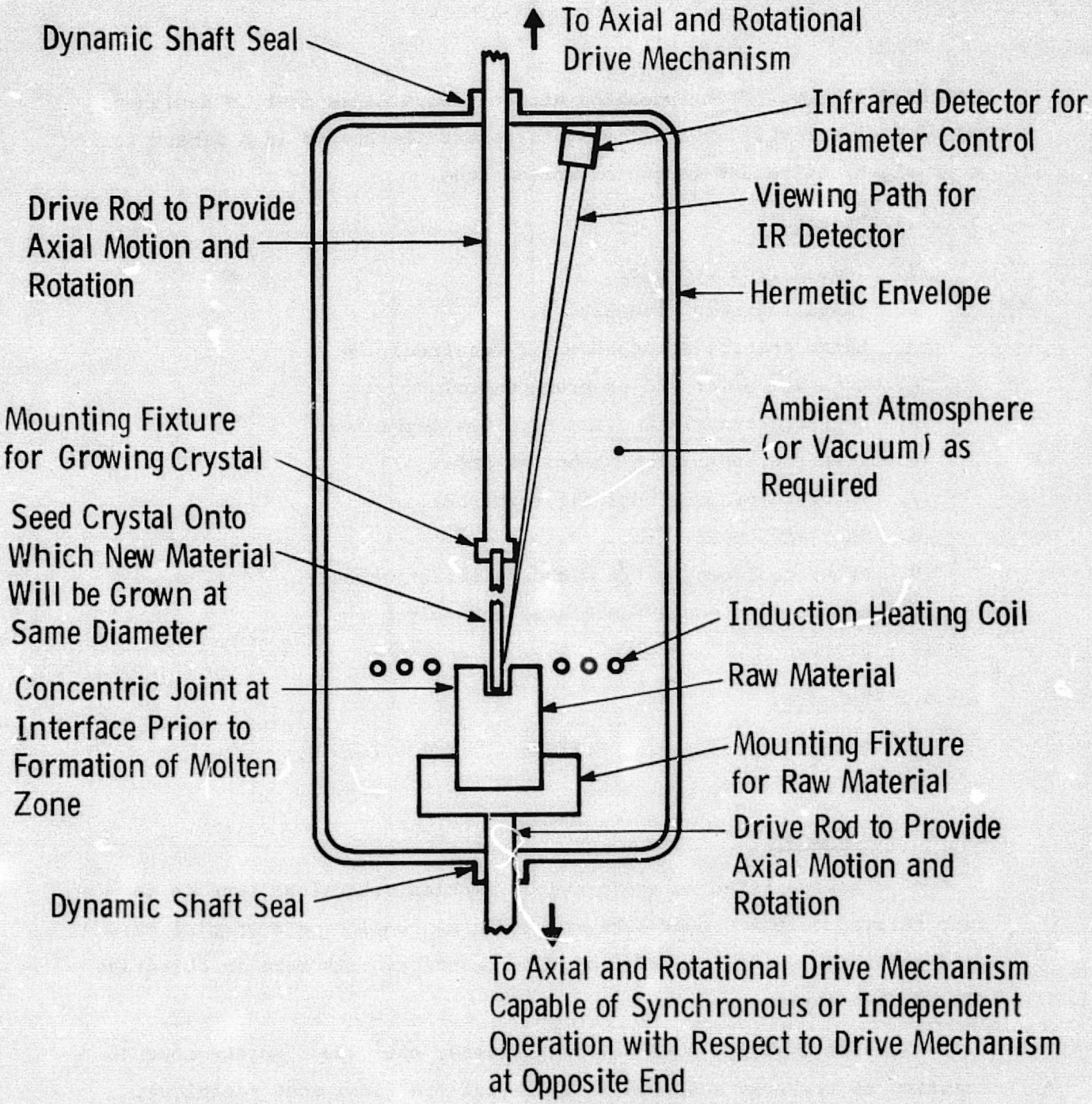

Basic mechanical requirements of diameter reducing molten zone method for automated control (Refer also to Fig. 3a)

Figure 4d

infrared energy. With induction heating, work coils must be designed, as with one-gravity applications, to direct the energy in a manner to strongly minimize the length of molten zone.

5.1.5.1 Procedure

1. Remove from stowage.
2. Install growth assembly.
3. Enter special atmosphere (if required).
4. Activate power and program control.
5. Activate translation and rotation mechanism.
6. Initiate separation of molten area.
7. Initiate program cool (if required).
8. Shut off power.
9. After cooldown, purge with ambient atmosphere.
10. Remove sample and associated assembly.
11. Stow.

5.1.5.2 Examples

A. Semiconductors, e.g. Silicon M.P $\sim$ 1450°C.
B. Refractories, e.g. YAG M.P $\sim$ 2000°C.

5.1.6 Float Zone

As indicated in the previous section, the float zone is in fact related to the Czochralski which can be considered a special case of float zone. Other examples of this technique vary more in objective rather than implementation.

For example, zone leveling whereby one uses a molten zone of solution to traverse a sample is essentially a float zone technique. In principle, a narrow zone is used with the advancing front dissolving material and the rear exsolving crystal. This is typically used in the growth of peritectics and solid solution crystals. Another application of this is the Traveling Gradient Zone Method.

Zone refining is a purification process identical in technique except for the fact that multiple passes of the zone are required for purification. Semiconductor crystals are frequently processed in this way.

The most frequently used apparatus for this process is to traverse a heater of restricted heating zone along a sample maintained at an elevated temperature. Alternatively, the sample may be moved along a stationary furnace assembly. The equipment description for this process is given in the Task 5 report, Bendix Aerospace Systems Division, NAS 8-30741. Of primary concern in this process is controlled traverse and heater zone temperature.

5.2 Electronic and Electrical Considerations

The purpose of this section is to discuss, in general terms, process control functions for "Shuttle" space flight materials processing experiments and also to present some conceptual ideas on control systems for these experiments. To begin with, it is virtually impossible at this time to identify specific control functions and parameters since specific experiments and associated furnace equipment are not yet identified. On the other hand, it is not unreasonable to discuss general classification of control functions and possible requirements of these based on knowledge of existing similar process functions. There are a number of different types of control functions anticipated for the Shuttle materials processing experiments. These include: (1) sensing functions, (2) power conditioning and regulation, (3) mechanical manipulations, and (4) process programming and control. Data recording is another very important system function even though it may not be part of any given experiment control loop. Each of these is discussed below.

5.2.1 Sensing Functions

There are three main types of sensing functions anticipated for the proposed materials fabrication processes. These include: (1) temperature sensing, (2) power or voltage sensing, and (3) position sensing of various types. The specific types of sensors or sensing functions used will depend greatly upon each specific process and associated process equipment. These sensors or sensing functions are critical parts of the process control systems because their accuracy and stability will greatly affect the processes in question. Output signals from such sensors are essential for the closed loop control of each process and also for data accumulation. Let us now examine more closely some sensors and sensing functions and discuss desirable operating characteristics.

5.2.1.1 Temperature Sensing and Signal Conditioning

Accurate and, more importantly, stable temperature control will be one of the most widely used control parameters. The temperature ranges of interest include 500°C to 1200°C for MA-010 type multipurpose furnaces, 500°C to 1600°C for platinum multi-zone type furnaces, and 1000°C to 2300°C for tungsten mesh heated and induction heated furnaces. The types of temperature sensors used in each case will depend upon the temperature range, accuracy and stability requirements, furnace design and atmosphere in which the sensor will operate. Thermocouples will certainly be leading candidates for the lower temperature processes and may also be very useful at higher temperatures if appropriate sensor atmosphere conditions exist. Optical pyrometer and light pipe-thermopile sensing techniques are likely candidates for the higher temperature processes. These techniques may suffer slightly in initial accuracy capabilities due to possible calibration shifts during spacecraft launch and transit, but can provide very stable and sensitive sensing functions. Care also must be taken in their application to

eliminate changes in their output signals due to contamination of their viewing lenses or windows.

There are two other operating characteristics of any temperature sensor that will permit maximum flexibility and usability in the process control systems. The first of these is electrical isolation of the sensor output signal from the electrical environment in which it is placed. The second is conditioning of the sensor signal to a level that is easily used and compatible with other system requirements. Both of these characteristics can be accomplished by appropriate electronic amplification and conditioning of the sensor signals. This is somewhat easier for the optical pyrometer and light pipe-thermopile type sensors because the sensing probes inherently provide excellent electrical isolation. Such is not always the case for thermocouple sensors, but techniques for providing the necessary isolation and amplification are well known and used in industry.

It is my opinion that temperature sensing functions for both control and data acquisition should be considered as modular functions where conditioned output signals (say 0-5 volts) are provided. This will enhance control system flexibility for both minor and major control loops and will automatically provide suitable signals for data acquisition, etc.

5.2.1.2 Power or Voltage Sensing

A second parameter that may be used for the control of a process is that of power delivered to the experiment apparatus. One may find that regulating power at a programmed rate provides more desirable process control than temperature sensing control. Furthermore, this can often be done by measuring the voltage applied to the heating mechanism and using it alone as the control parameter. This is often the accepted industrial practice when induction heating is used. As in the case for temperature sensing functions, it will be

desirable to derive voltage or power signals (perhaps 0-5 V_{dc}) to simplify integration into the system. Signal levels will probably never need amplification and, where ac power control is used, electrical isolation will be easy to achieve by appropriate use of transformers. Special signal conditioners may be desirable to yield dc signals corresponding to applied RMS voltage where waveshape irregularities exist. In the cases where dc voltage is used for the heating elements, special isolating techniques such as those used for thermocouples may be desirable.

5.2.1.3 Position Sensing Functions

Special position sensing functions may be required for control of specific processes. The simplest of these may be a limit switch indicating the position of some linear motion for a given process. A much more complicated one would be the position sensing of the growth interface in a Czochralski process. Laboratory experiments using a focused infrared detector have proven to be feasible for such a function. It is easy to visualize that the exact nature of any position sensing devices will depend upon experiment and furnace apparatus identification. Again, as discussed earlier, it will be most desirable to adhere to some set signal condition standards for the output signals have such position sensors.

5.2.2 Power Conditioning Functions

The power conditioning functions required for the different processes and furnaces must be identified and developed very carefully. For purposes of simplification, let us limit this discussion to the power conditioning equipments that directly provide power for heating of the various furnaces or heating of the process workpiece directly. Let us consider the inputs to be the power busses and a control voltage signal (say 0-5 V_{dc}) and the output to be a regulated power or voltage

proportional to the control signals. The types of heating elements that will be required for the various processes include resistive heaters (both constant resistance and highly temperature dependent resistances), induction heaters, and possibly optically focused heating and electron beam heating. Power conditioning equipment to drive such heating elements at proposed power levels are readily available for standard industrial applications.

It is evident that the power control concepts utilized in these equipments can be used for the proposed Shuttle materials processing equipment. However, it is also evident that such power conditioning equipments will require at least minor or, very likely, major modifications to be acceptable for the Shuttle missions. There are a number of factors that must be considered in determining the feasibility of these power conditioning pieces of equipment. The following paragraphs are an attempt to identify major items of concern and to discuss briefly how they will affect selection or development of suitable power conditioning equipment.

5.2.2.1 Size, Weight, Packaging Considerations

Perhaps the most obvious set of considerations which will affect the utilization of given power conditioning equipment are those related to its physical characteristics. Commercially available hardware is generally designed for quite different operating conditions than those that will be present in the Shuttle spacecraft. They are designed to operate from 115 or 230 V_{ac}, 60 Hz power sources and often have large transformers and large air-cooled heat sink areas. At the very least, modifications would be necessary to accommodate either 115 V_{ac}, 400 Hz or 28 V_{dc} input power source and, also, to accommodate the different cooling properties available in the spacecraft. Even then, considerable repackaging may be required to reduce the physical size

of each power converter (thus permitting maximum use of available space) and also to make the equipment rugged enough for launch acceleration and vibration conditions.

5.2.2.2 Power Source Loading Considerations

The power conditioning equipment will have to be regulated to prevent overloading of the available spacecraft power buss. Considering the power levels that will be required (2-5 KW) per furnace, techniques to limit the power drawn from the power buss will be most desirable features in each piece of power conditioning equipment. This will be particularly true for such loads as platinum or tungsten resistive heaters. Current limiting techniques are presently used in commercial equipment and may need only slight modification to meet the limits that will be set for the spacecraft buss supplies.

5.2.2.3 Efficiency Considerations

The consideration of efficiency in power conditioning equipment encompasses a wide variety of factors each of which can greatly influence system efficiency. To begin with, high efficiency in the power conditioning equipment is very desirable because it can greatly reduce the size and cooling requirements. In turn, this reduces the demands on the spacecraft power busses whether one considers the 28 V_{dc} fuel cell buss or a possible 115 V_{ac}, 400 Hz power buss. Let us look at some factors that can influence efficiency.

The most efficient types of power conditioner are those that incorporate some form of Class C switching control technique to regulate the output power. By use of chopper regulation, phase-shift modulation of applied ac power, or some other similar technique, power conversion factors of 90% or better can be achieved. On the other hand, Class A type controllers could easily have efficiencies less than 50%.

Another factor that may be controlled by proper design is that of matching the load to the power source. In some cases, especially the MA-010 type furnace where constant resistance Kanthal heater elements are used, the heaters may be designed to permit a close match to the 28 V_{dc} buss. In other cases, where heater resistances vary drastically with temperature or cannot be made to match the 28 V_{dc} buss power source, appropriate transformer design and operation from the 115 V_{ac}, 400 Hz buss can provide good power source-load matching. Such load matching considerations will enhance power conditioner efficiency and minimize worst case disturbances on the power buss lines.

5.2.2.4 RFI-EMI Compatibility

Another item that will require much closer attention for Shuttle application vs commercial application is that of RFI-EMI compatibility. This can be particularly true for power conditioning equipments since they can generate considerable interference if proper attention is not paid to this problem. Special precautions must be taken with the use of induction heating to eliminate interference in other processing equipment. The use of Class C switching techniques for other power regulation functions (desirable from a viewpoint of maximizing efficiencies) must also be done with care to meet expected RFI-EMI compatibility standards. These standards could be very strict if they follow those set forth for Skylab and Apollo-Soyuz missions. On the other hand, it may be possible to relax many of these standards considerably if a fuel-cell power source system is designated specifically for the materials processing experiments. However, it is still expected that stringent standards will be required for any interfacing with other Shuttle systems, such as data acquisition and telemetry interfaces.

5.2.3 Mechanical Manipulation Functions

Several types of automatic mechanical manipulations functions are anticipated for the various materials processing experiments. These

functions are characterized by low power requirements and a need for very stable controlled rates of mechanical motion that can be varied over wide ranges. Both linear motion and rotational motion functions are common. Commercial equipment for performing these functions usually includes small precision dc motors and appropriate gear assemblies to get the desired motion. Precision tachometers are usually coupled to the motor shafts to permit sensing and closed loop control of motor speed in response to a dc voltage control signal. Adaption of these control techniques to Shuttle materials processing equipment should be relatively easy.

5.2.4 Data Recording and Transmission

The recording and transmission of process data is essential for complete analysis of the results of each experiment process. The number of pertinent data points is estimated to vary between 2 and 10 points depending upon the given experiment. Fortunately, the required data recording rate can probably be very slow (i.e., each point being samples once every 1-5 minutes). The types of data that will be desired will be process temperatures, power levels, pull rates, etc. Time correlation for each data point is also required. Pertinent environmental data, such as spacecraft accelerations should also be recorded for each experiment.

Data acquisition, storage, and telemetry systems such as those used in previous space missions have more than enough storage and telemetry capacity for the needs of the anticipated materials processing experiments. However, some arguments may be made to increase the bit capacity of the system to permit at least 10 bit conversion accuracy of the various process parameters. This is particularly true for temperature data. Previous 8 bit conversion standards would limit conversion accuracies and data resolution to approximately $\pm$ 10°C for temperature measurements of 2200°C.

Interfacing of the process parameter signals with the data acquisition system should not present any problems as long as signal conditioning and isolation standards such as those discussed for the sensing functions are followed. In fact, standards for those process signals should be developed around interface requirements for the data acquisition system. Should an on-board computer be used to operate the various experiment processes, the data will probably already be in a format directly usable for recording and transmission. Of course, the burden of interfacing with the process analog signals then is simply transferred to the computer input interface.

5.2.5 Process Programming and Control Functions

Process programming and control functions for the various experiments will have many similar characteristics. Typical process programming functions include: (1) start of process, (2) set process soak temperature (temperatures) or power levels, (3) set pull or rotation rates, (4) set controlled cooldown rates, and (5) system shutdown. All of these are programmed as a function of time or feedback information from the process. It is very desirable to automate these functions as much as possible and, at the same time, provide a wide range of variation in all parameters to facilitate flexibility. Possible concepts of automated programming and control functions are discussed later.

Automatic control of the different process functions for each experiment will be performed by controllers in conjunction with the power conditioners, mechanical actuators, etc. Operation of the controllers will normally be in response to their respective input program signals and corresponding feedback signals from the process equipment. Their primary function will be to generate error signals which automatically adjust the output functions (power, voltage, motor speed) to meet the demnnds of the process programmer. Sometimes, special operations such as error integration and error gain will be incorporated in the control functions to guarantee accuracy and stability.

Output current limiting is another special function that may be incorporated in certain controllers. At other times the control functions will be simple enough to operate "open looped" directly in response to the program signals and will require no process feedback information. Compatibility of input and output signals of the controllers will be part of the total system design requirements.

5.2.5.1 System Control Concepts

A wide variety of system control concepts can be developed for the various material fabrication processes. To a certain degree, these will be governed by the processes and functions to be performed. On the other hand, careful consideration in the design of specific experiments can overcome limitations of the process control system. In either case, the final choice of a system control concept will require consideration of both the experiment processes and available control techniques. The discussions in the previous sections have been oriented toward describing specific types of functions and setting characteristics that will facilitate system development and flexibility. Let us now examine some possible control system concepts.

One control system concept that is certainly feasible for these processes is that employed in the Skylab M518 Multipurpose Furnace System and the Apollo-Soyuz MA-010 Multipurpose Furnace System. This type of system makes use of a specially designed control system to operate with a specific furnace assembly. A block diagram of this materials processing system is shown in Fig. 5. The control package provided means for manually setting various program parameters such as soak temperature, soak period duration, and controlled cooldown rate. It also provides the required power conditioning function to control the furnace temperature in response to the set program and thermocouple temperature signals from the furnace. Amplified and isolated thermocouple voltage signals were also generated for telemetry purposes.

A similar control system concept could also be employed for each experiment process in the Shuttle mission. In many cases,

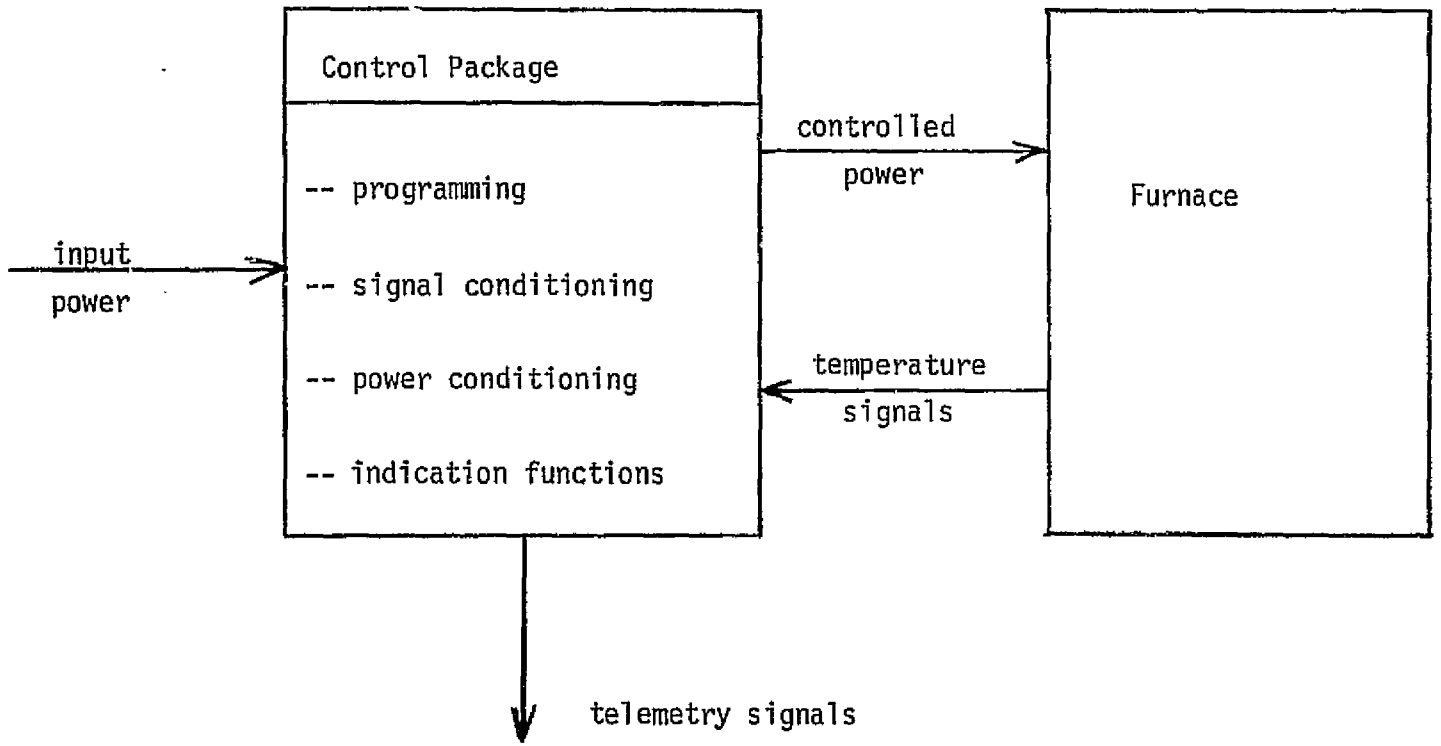
Control Package
-- programming
-- signal conditioning
-- power conditioning
-- indication functions
input
power
controlled
power
Furnace
temperature
signals
telemetry signals
(0 - 5V dc)

Figure 5

additional parameter sensing and control functions would have to be incorporated in the control package. Such a control system concept could be technically feasible and probably require a minimum amount of dependence on other spacecraft equipments for operation. However, this conceptual approach would probably require special control system design for each experiment process and probably decrease the total system flexibility.

A second control system concept would make extensive use of an on-board computer for programming and control of each experiment process. The computer would directly interface with all process power conditioners, mechanical actuators, and process sensing devices to provide the necessary control functions. A block diagram of such a process control concept is shown in Fig. 6. The main advantage of this type of system concept is that the use of a computer for programming and control functions can provide great system flexibility. It also would eliminate the need to design special hardware for each process. A limited number of different power conditioner modules could probably be designed to cover all different heater requirements. On the other hand, there are certain control functions which the computer may not be able to perform very well. One of these is current limiting control in the power conditioner hardware. Also, the inherent discrete bit characteristic of the computer might seriously limit the ability of system to achieve very stable, continuous control of some process functions. It could be extremely difficult, if not impossible, for the computer to provide the continuous linear control function that will be required for many temperature and power regulated process functions. Thus, it appears that a system concept based on performing all of the control functions in a central computer also has serious limitations.

Some modification of the described computer control concept would yield a more desirable control system concept and provide more stable process control functions. Certain control functions can best be done by incorporating minor loop control techniques. This is

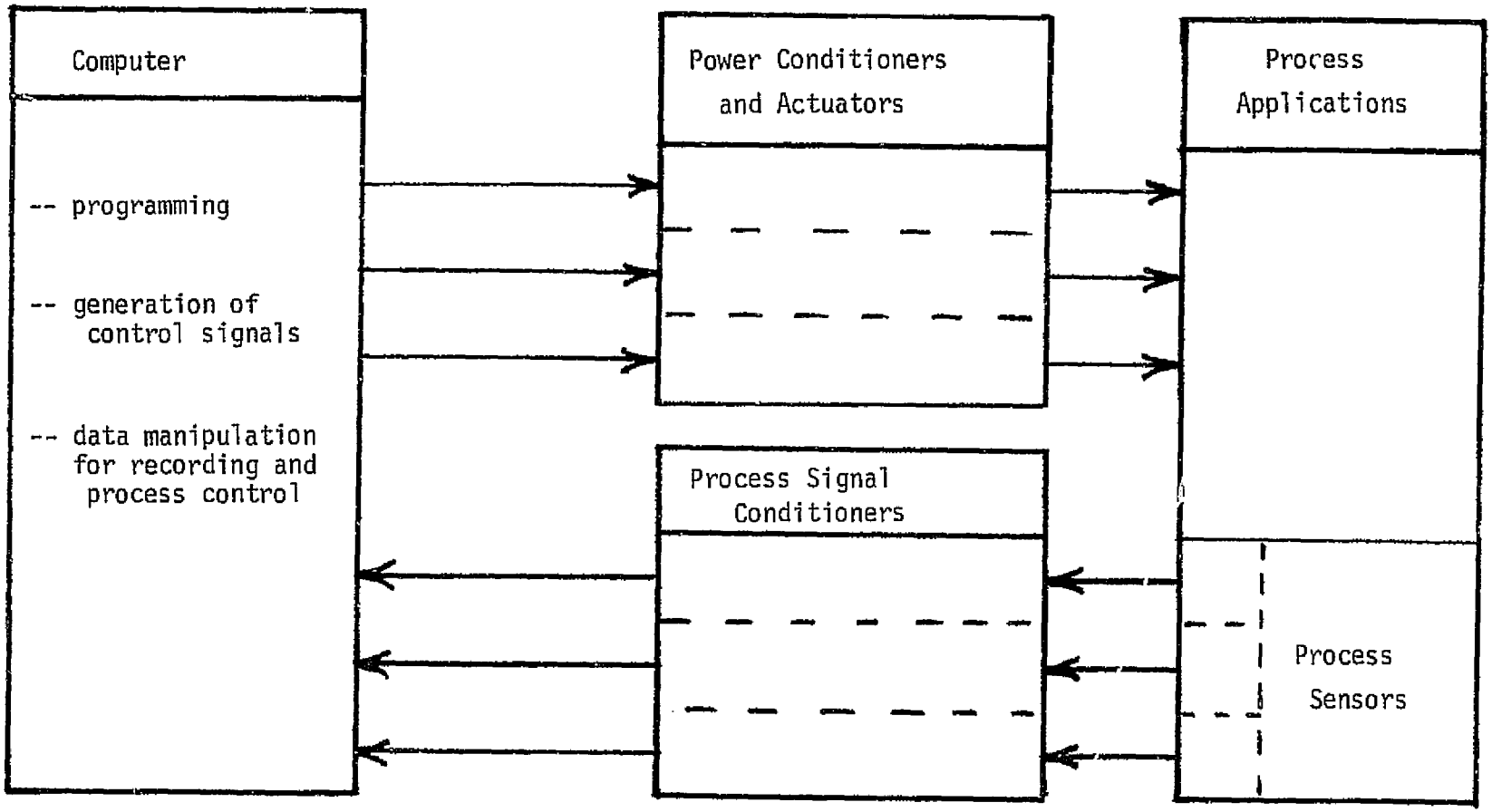
Computer
-- programming
-- generation of control signals
-- data manipulation for recording and process control
Power Conditioners and Actuators
Process Signal Conditioners
Process Applications
Process Sensors

Figure 6

particularly true for high resolution power and temperature control functions. A block diagram illustrating the resulting system concept for one operating function (i.e., temperature control) is shown in Fig. 7. In this example, additional control requirements are placed in the power conditioner where they can be more effectively performed. Programmed set point information is delivered to the power conditioner module from the computer. Analog temperature feedback information is also fed back to this module. Error sensing and amplification functions can then be incorporated in the power conditioner module to provide the desired continuous control function to assure process temperature stability. In addition, a current limiting function can also be incorporated directly in the power conditioner circuitry.

One might also note that this block diagram shows two control set point input signals to the power conditioner. It is proposed that one of these provide a "coarse" resolution temperature signal and the other a "fine" resolution temperature signal. The resultant input signal to the power conditioner would be the sum of these two signals. A typical application might place a "weighting" factor on the "fine" control signal of 1/10 that placed on the "coarse" control signal. This technique will permit much better resolution set signal capabilities from the computer (limited by the discrete bit characteristic of the computer) and possibly enable the computer to generate very fine resolution programmed cooldown functions it otherwise could not do.

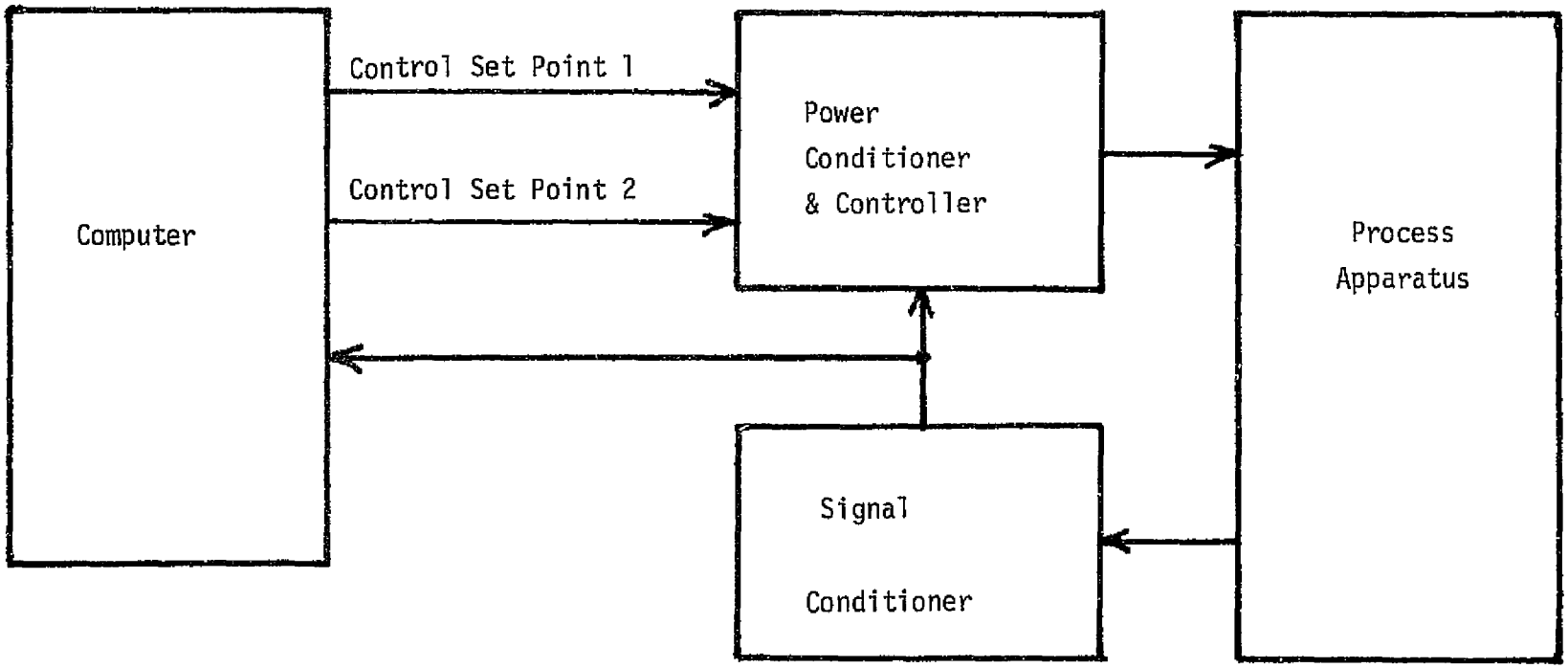
Computer
Control Set Point 1
Control Set Point 2
Power
Conditioner
& Controller
Process
Apparatus
Signal
Conditioner

Figure 7

5.3 Furnaces

Although it is obvious that experiment requirements will define the furnace capabilities made available, it seems desirable to present a conceptual configuration which would indicate a broad range of options. There has been no attempt made to maximize the number of furnaces or to develop any preferred orientation for furnace unloading or loading. Instead we have chosen to provide a facility that is capable of performing the experiments described earlier that will enable one to estimate power, volume, and sample size in a given facility.

Such a conceptualization is shown on Fig. 8. We have elected to have all furnaces loaded and unloaded (as required) from a single direction. The furnace subelement consists of three basic segments, each having associated liquid cooling integral to the heat sink: a bank of 5 MA-010 types, a center segment consisting of a platinum resistance and tungsten mesh heater furnaces, and a facility frame provided with translation and rotation mechanisms opposed to one another that can work synchronously or independently of one another. The third segment contains an enclosure (cooled) in which may be mounted a spherical reflector and lamp imaging heater, leadthroughs for an RF coil or coils, or a tungsten mesh furnace. The total volume element including these facilities will require approximately 1.5 cubic meters exclusive of cabling and cooling lines. As a result, the furnace subelement circumscribed volume of 2.8 cubic meters will be less than 2/3 utilized leaving adequate volume for simple automation equipment.

5.3.1 MA-010 Furnace Bank

This element is conceived as comprising 5 furnaces, 3 with triple cartridge accomodation and two with a single cartridge facility. These are Kanthal wound with a maximum continuous temperature at the hot end of 1150°C. Those experiments that require a nearly isothermal environment and are located in the hot end can be run at the maximum temperature. Those samples that require a gradient freeze wherein

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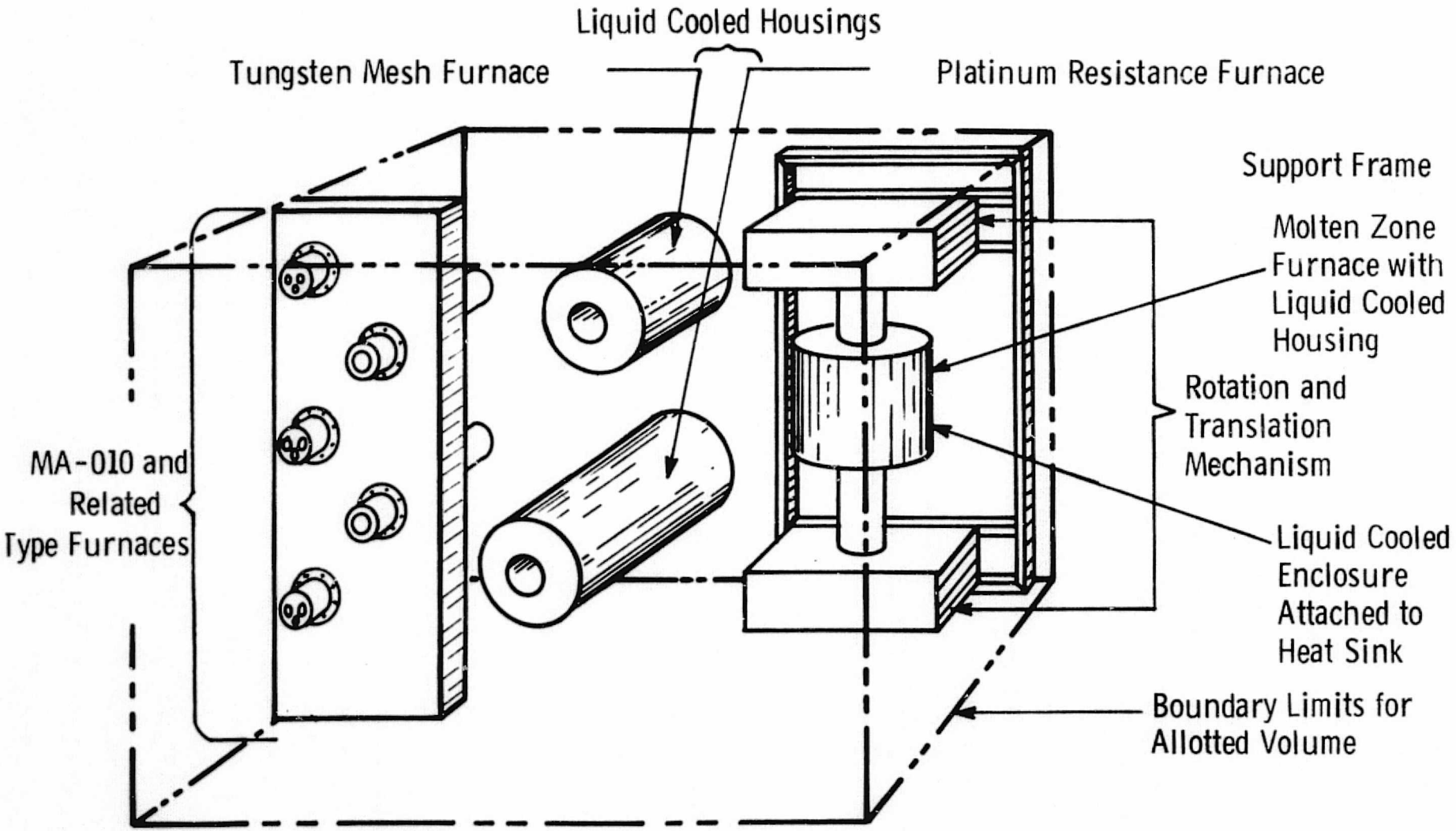


Example of feasible furnace sub-element arrangement

Figure 8

melting is accomplished by longitudinal heat flow along the sample must have a maximum melting point of 1000°C in order to obtain meltback and a temperature gradient. The cartridge volume for containing sample in a gradient experiment 8 cm in length is about 15 cm^3 per cartridge or 45 cm^3 in a three cartridge furnace. This indicates an experiment sample volume (exclusive of thermal insert) of approximately 25 cc. In the isothermal region of the furnace approximately 10-20 cc of experiment material can be accomodated. In the single cartridge furnace sample material having a volume of 35-45 cm^3 can be processed in the nearly isothermal region. Although power consumption is a function of operating temperature, thermal conductivities of cartridges, and gradient requirements, a fair estimate of total power consumption would be 200 watts maximum/furnace.

Experiments	Max.Power (watts)	Sample Vol cc
1. Three vapor transport	150	5
2. Three gradient freeze (Germanium)	180	25
3. Three gradient freeze (eutectics)	200	25
4. One isothermal solution growth (1150°C) (Solvent freezes in situ at run termination)	180	40
5. One isothermal melting (metal alloy) MP 1150	180	30

5.3.2 Resistance Heating Bank (Pt, W)

Both the platinum wound and tungsten heater furnace are commercial equipment. The former has multiple taps as a controllable gradient at temperatures up to 1500°C (continuous operation at 1400°C). The working volume will vary dependent on the experiment parameters but a 40 cm long furnace with a 6 cm bore is capable of processing a sample 150-200 cm^3 in volume with a power consumption of between 1-2 kW at 1100-1400°C. The tungsten mesh furnace can process volumes up to 500 cc (using the total heating zone) at 2200°C at 5-6 kW.

	Experiments	Power Watts	Vol cc
(Pt)	Directional solidification (silicon, Fe, Cr, Ni alloys, eutectics)	1500	150
(W)	Isothermal solidification (2000°C)	> 5000	250
	Solution growth (1400°C)	< 2000	500
(W)	in conjunction with frame facility		
	Costing	< 5000	100
	Solution growth (seeded)	< 5000	200
	Czochralski (2000°C)	> 5000	200

Power estimates of the tungsten furnace in conjunction with the frame assembly can be estimated only crudely since the efficiency of insulation associated with the use of translation and rotation motion mechanisms cannot be determined without detailed design concepts.

5.3.3 Frame Assembly

As mentioned above, heating can be performed with the tungsten resistance heater. R.F. heating and power requirements are discussed in Bendix task report. The final heating mechanisms to be considered are electron beam and imaging. Electron beam heating like RF requires special equipment external to the furnace subelement and specifications other than Skylab equipment are not available. Commercial imaging furnaces are available and estimates of performance are possible. Equipment using a 1 kW tungsten-halogen lamp can perform float zone experiments on 0.6-1 cm diameter rods having melting points at 1600°C. Higher rated lamps with commensurately higher temperature and sample size capabilities can be considered.

5.3.4 Improvement of Furnace Performance

The examination of the normal operating characteristics of two laboratory furnaces were examined and possible methods for upgrading performance were considered. The two furnaces, a tungsten mesh heater,

high temperature unit and a medium temperature platinum wound, multi-element tube furnace were representative of typical high quality furnaces used for materials studies in many laboratories. The tungsten mesh furnace uses molybdenum radiation shields for thermal insulation whereas the tube furnace uses a more conventional semi-solid insulating material. Some reduction in power requirements should be possible in both furnaces with only minor changes in design or construction; whether or not such changes are attractive for space flight applications depend on the nature and capabilities of other system interfaces. Nevertheless, the improvement in efficiency should be rather easily accomplished in the main and would seem expedient even if not mandatory.

The first subject for study was the tungsten mesh furnace which is capable of operation to about 2400°C in vacuum. The power/temperature characteristic of this furnace when operated in vacuum can be well represented by the empirical equation $P = 2.4 \times 10^{-13} T^4$ indicating that radiation is the main heat loss mechanism, although there must certainly be losses by conduction via the water cooled power leads. Neglecting this conductive loss for the moment, the effectiveness of the shielding can be estimated from the P vs T^4 equation. The heater is about 10 cm in diameter by 15 cm high for a total surface area of about 630 cm^2. At 2273°K, the radiative power loss from a block body would be 151 W/cm^2 whereas the furnace loss would be about 10 W/cm^2 for an effective shield emissivity of 0.067. The anticipated value for a set of six molybdenum shields would be about 0.025 for the effective emissivity assuming $\varepsilon \approx 0.3$ for molybdenum.

If the conductive heat losses from the furnace were included, the observed emissivity of the shield assembly would be reduced somewhat. These conductive losses should not, however, be a large fraction (e.g., 0.5) of the total losses, so one must conclude that considerable improvement in operation could be achieved by improvement of the shield design. The total number of shields could easily be tripled by the use of molybdenum foil interstitial shields between the heavier main shields. This should reduce the power lost by radiation to a third of its present

value and hence the total power by about one half. Additional, albeit minor, improvement could also result from careful fabrication of the shield assembly to reduce radiation leaks. All of these changes could be accomplished with very minor changes in the overall design of the furnace.

The tube furnace analyzed for this study has a hot zone 6.35 cm (2.5 in.) diameter by 40.6 cm (16 in.) long. Its power/ temperature are more linearly dependent on temperature as would be expected from the solid insulation used in its construction. The calculated conductivity of that insulation is about 0.002 W/cm-K which is typical of many fibrous materials. As with the tungsten mesh furnace, some improvement in power efficiency could be achieved by improving the furnace insulation. There are several materials on the market which have conductivities in the range of 5 to 8 x 10^{-4} W/cm-K and could be used to reduce the heat loss from this furnace. Unfortunately, most of these materials tend to degrade in mechanical properties at the maximum operating temperatures of a platinum heater furnace, so that a multiple layer construction would be necessary to benefit from their excellent thermal properties. A somewhat less efficient but more refractory insulation would be used adjacent to the heater elements while the more efficient but less refractory material would be used as the outer blanket.

Finally, some additional improvement in furnace performance would result from operation in vacuum rather than in a gaseous ambient. Unfortunately, this would probably somewhat lower the operating temperature, due to degradation of the filament but it would reduce the heat losses from the apparatus.

Unfortunately, the data available for analysis is inadequate for precise evaluation of what improvements could be expected. An estimate, however, would place the improvement somewhere between 25% and 50%. It is felt that this could be accomplished with relatively little impact on the cost of the apparatus and on its physical character such as size, weight, etc.

5.3.5 Furnace Specification Summary

TABLE 1

	wt (kg)	Vol. (cm^3)	Max. Temp °C	Max. Power (kW)	Sample Volume (cc)
*MA-010 (Single)	5	500	1150	0.1	45
MA-010 (triple)	5	500	1150	0.1	25
Platinum	20	20,000	1500	4	200
Tungsten	25	10,000	2400	10	500
Lamp Imaging	25	700	1700	2	(Zone) 1

*Total power is sum of listed power + sample heat loss.

TABLE 2

	MA-010	Platinum 4 Shunt	Platinum 8 Shunt	Tungsten (Gas)	Tungsten (Vacuum)
900°	.06	.6	.7 kW	2.1	1.0 (.6)
1500°	--	1.9 (1)	3.8 kW	3.2	1.8 (1)
2000°	--	--	--	8.0	6.0 (3)

These data show an indication of the furnace specifications and performance. The data listed give approximate figures for power consumption of different options. MA-010 power data is for the furnace alone. Since the experiment sample is also a path for heat flow, the total power consumption is the sum of furnace losses and losses through the sample. The figures in parentheses in Table 2 are projected power consumptions with minor modifications to the commercial furnaces. These do not require changes in the envelope or major changes in basic design. Previous discussions concerning power consumption have been based on estimated powers of unmodified furnaces. True potential power consumption should be reduced accordingly if modifications to equipment are anticipated.

5.4 Flight Worthiness

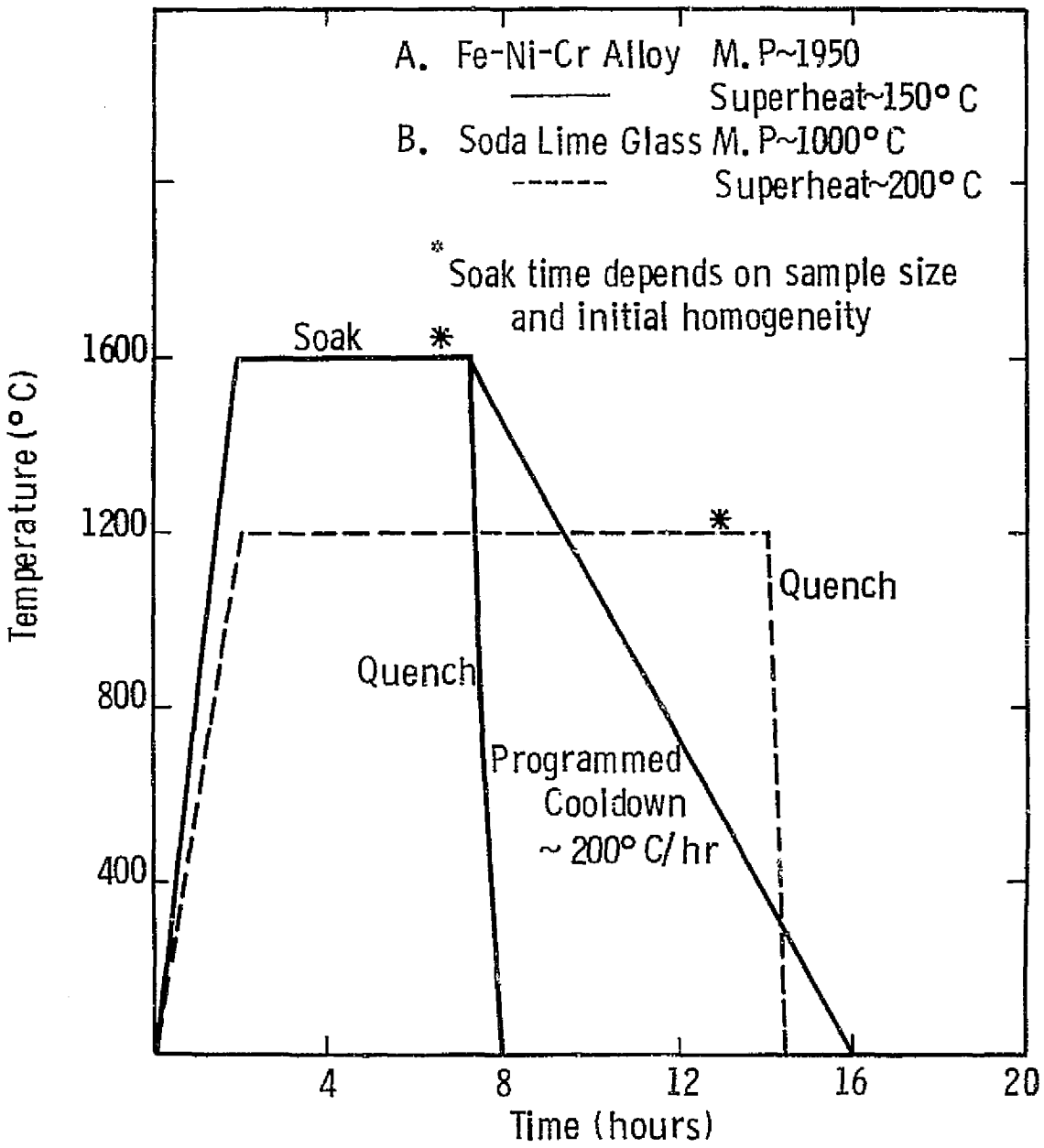
A superficial analysis was made on the suggested furnaces. The MA-010 and platinum resistance furnaces are satisfactory and should withstand launch loads. The lamp imaging assembly appears able to withstand loads. The tungsten-halogen lamp should be stowed separately and installed when ready for use. This is a relatively easy procedure.

Some problems can be foreseen with the tungsten elements. They are not firmly mounted and tungsten heaters are brittle and may be damaged during launch. Stowage of the heaters and installation in orbit is a substantial effort with the present configuration. As a result, significant redesign may be required. Alternatively, a mechanism for cushioning and supporting the heaters may be devised. One can make conjectures how this may be done, although it is clear that experiment and analysis will be required. For example, one can conceive of using a material with a low sublimation temperature for packing the heaters. Before use a vacuum is drawn and low heat applied to evaporate the packing material. Several organic compounds could qualify but some concern of flammability and toxicity have to be considered. A thorough evaluation of brittle heaters will have to be considered before selecting this type of furnace for flight.

5.5 Experiment Timelines

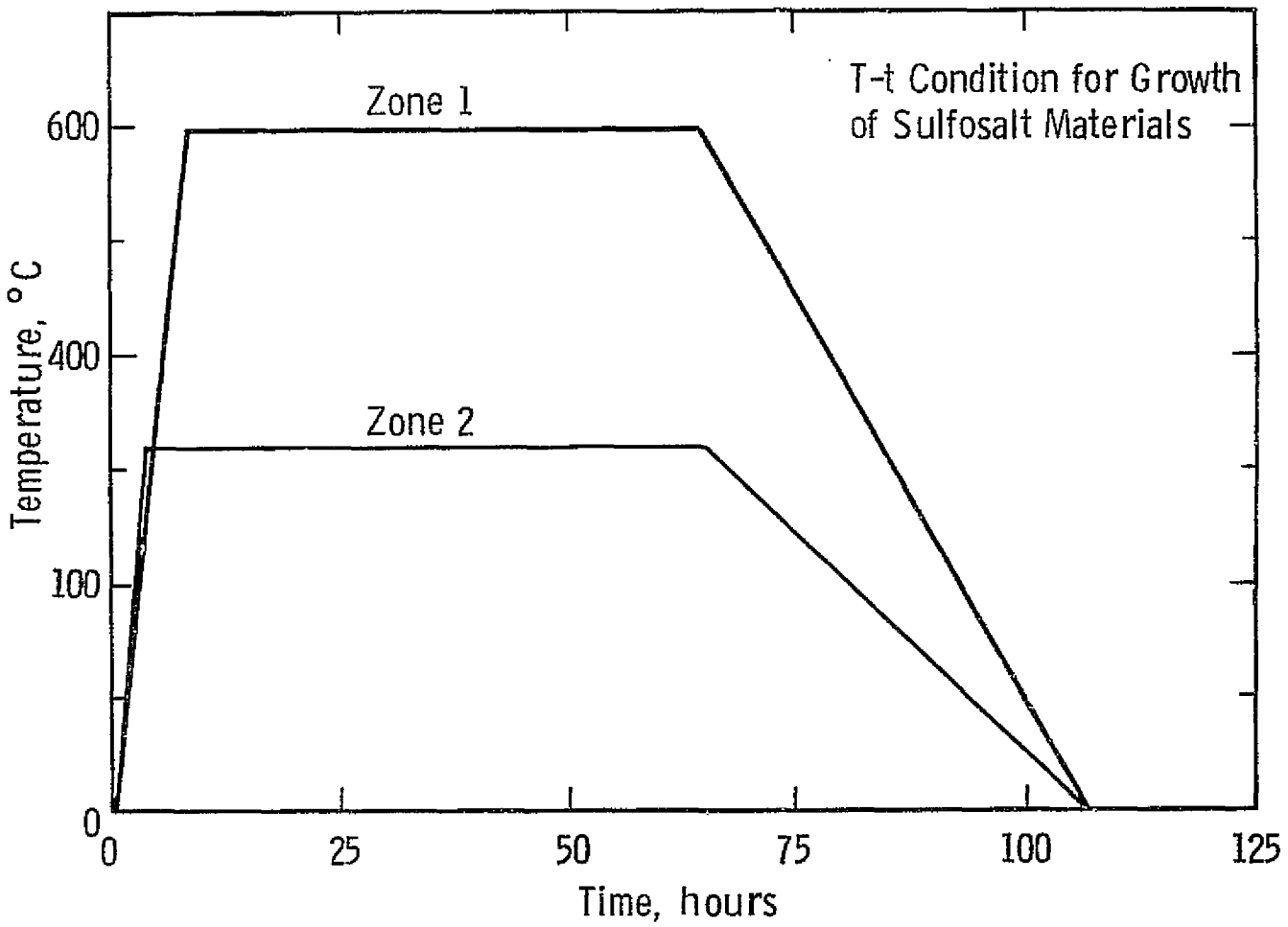
The following figures show some sample timelines of what may be considered typical experiments. These are presented only to indicate order of magnitude times which can be converted, if desired, to power consumption. Thus, heat-up times will be intervals of full power, experiment processing requires reduced power appropriate to the particular furnace or heating mode selected, and cooldown may be controlled cooldown at power or passive cool with furnace off. The experiment times will vary with material system, experiment goals, and the temperature selected and as a result will vary significantly. For example, sulfosalts

Curve 679145-A



Melt casting experiment examples

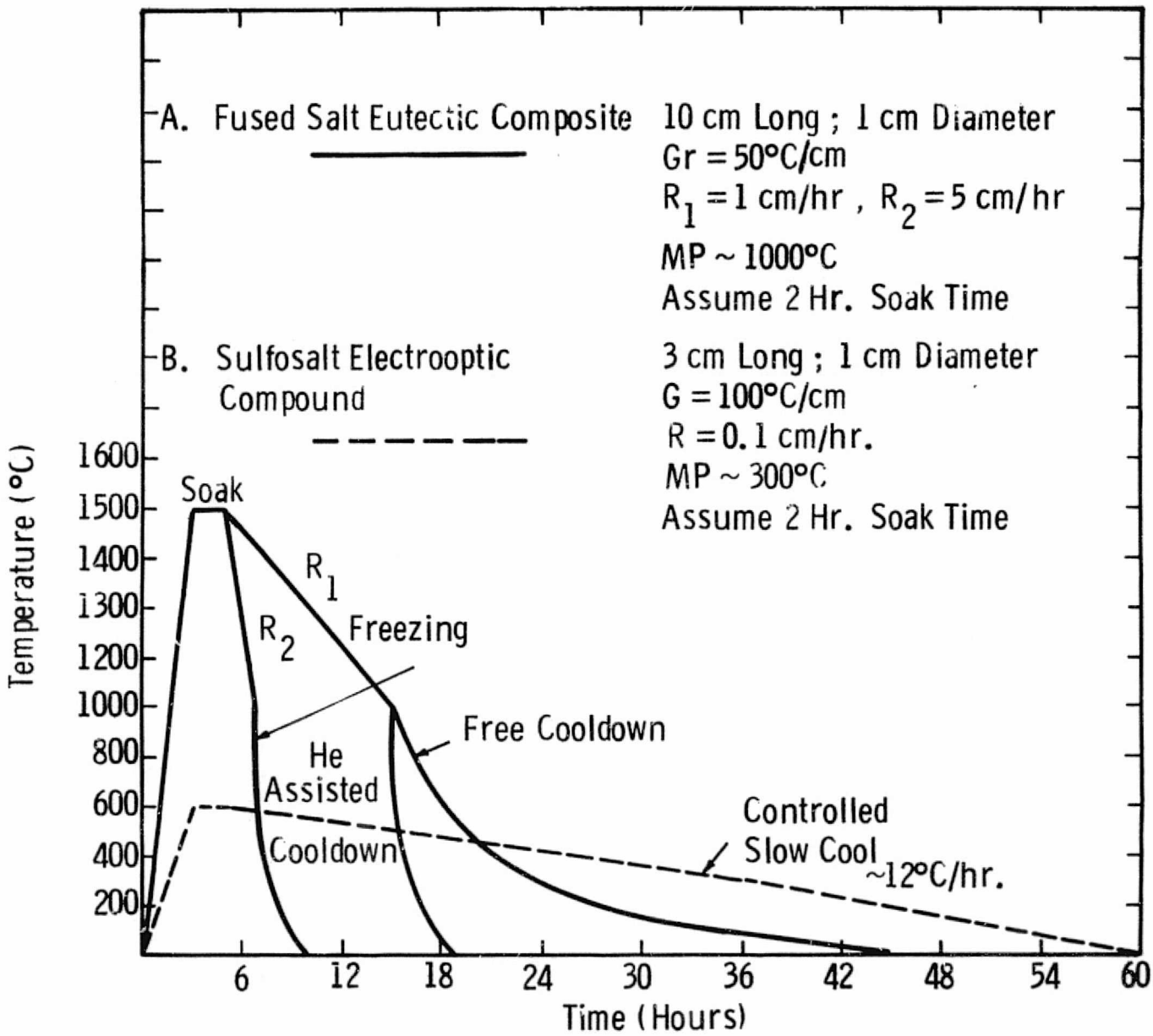
Figure 9



Bridgman crystal growth: two-zone furnace

Figure 10

Curve 679146-A



Examples of gradient freeze experiments

Figure 11

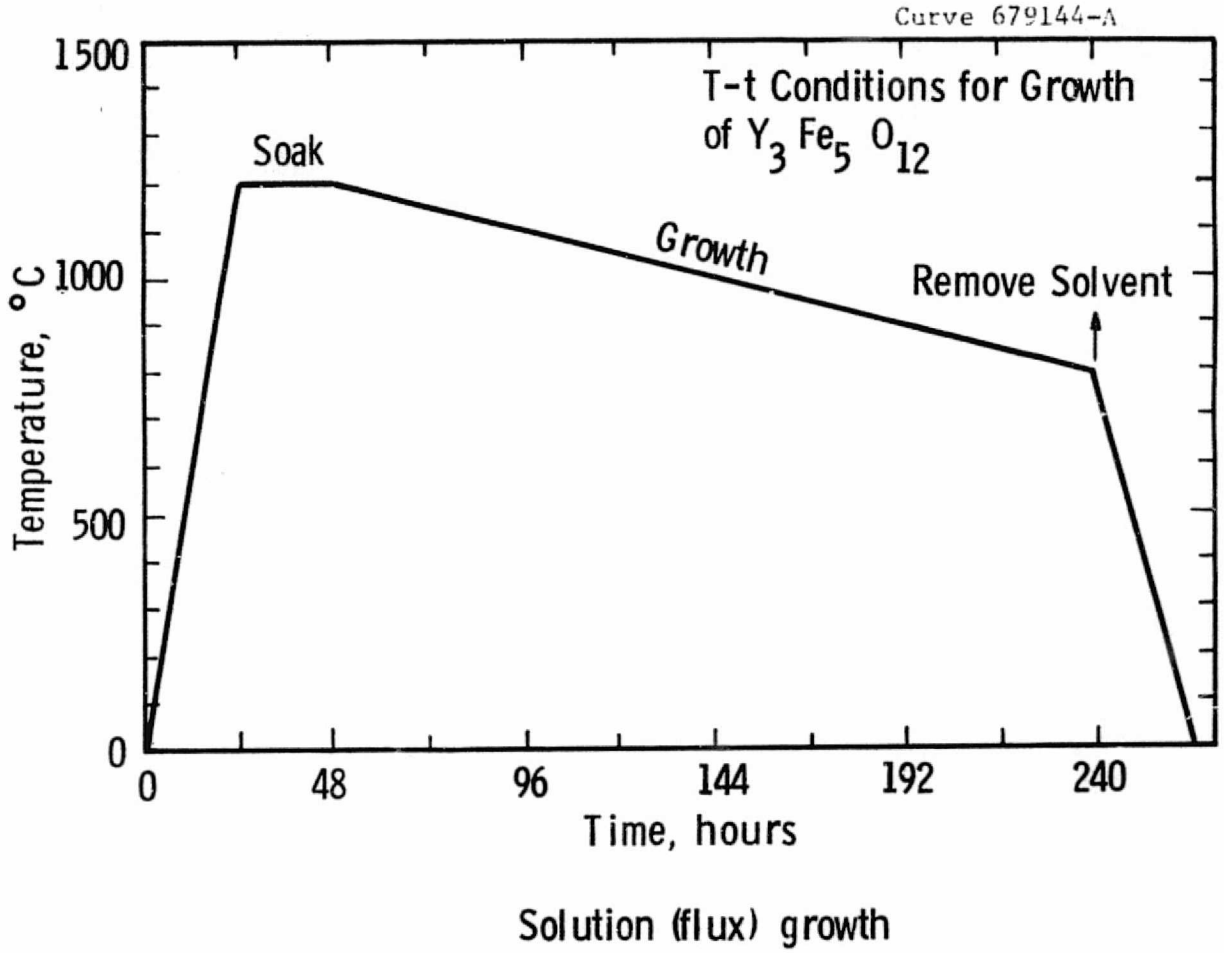
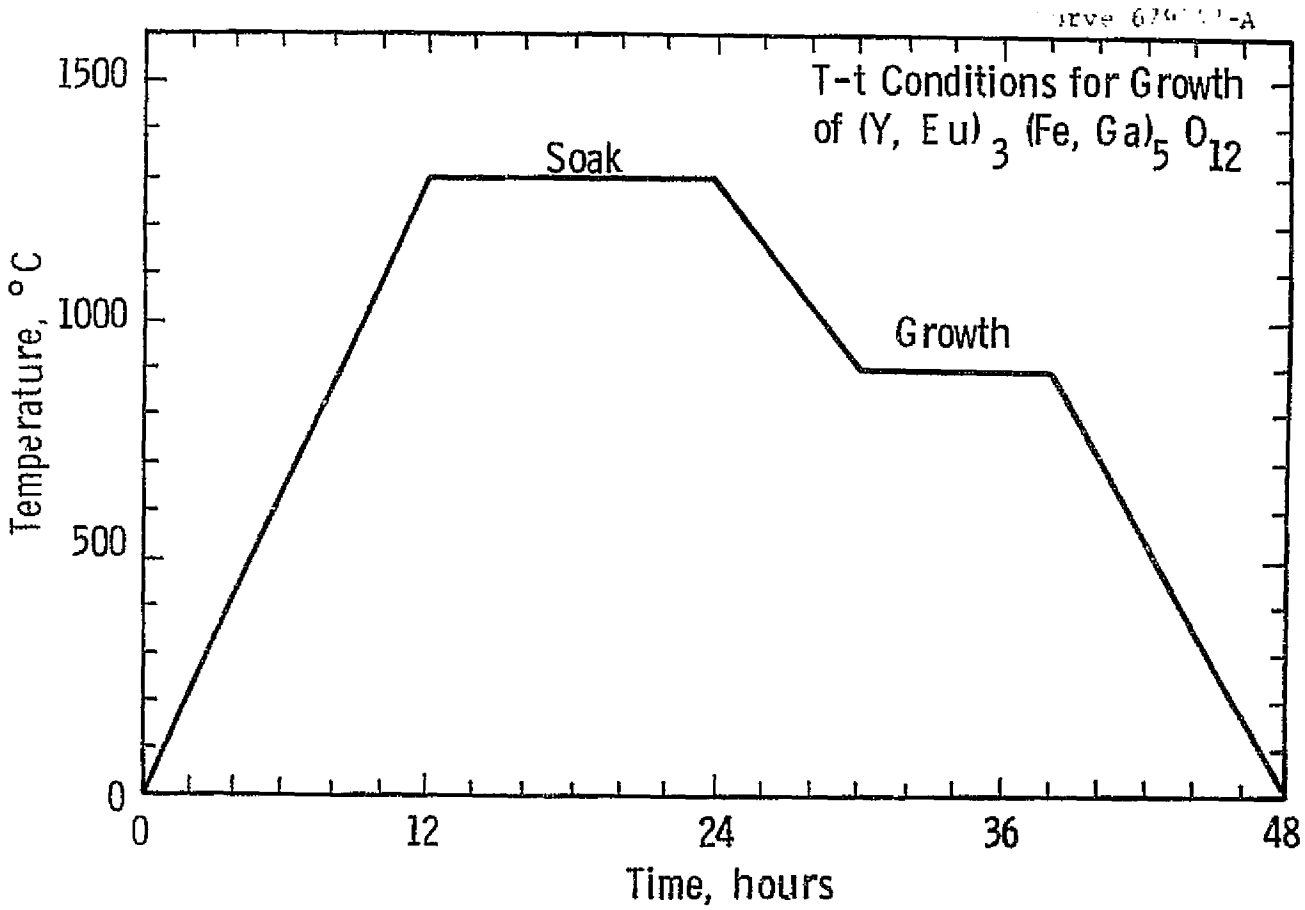

Curve 679144-A
T-t Conditions for Growth of $Y_3 Fe_5 O_{12}$
Soak
Growth
Remove Solvent
Temperature, °C
1500
1000
500
0
0
48
96
144
192
240
Time, hours
Solution (flux) growth

Figure 12



Liquid phase epitaxial growth

Figure 13

require slow solidification rates while metal eutectics can be solidified rapidly. However, there is a wide variation possible if, for example, the experiment involves impurity segregation phenomena or interface breakdown. Therefore, the timelines should be used as indicators of process requirements and not used for detailed planning.

6. STUDY LIMITATIONS

The primary limitation of a study of this type is a lack of detailed knowledge of the chemical systems that are to be run. For example, we can look at a straightforward Czochralski experiment. Neodymium-doped yttrium aluminum garnet by virtue of a low segregation coefficient must be grown at a rate of 1-2 mm/hour. Undoped silicon in the same apparatus may be run at 30-100 mm/hour in the normal mode while silicon ribbon growth can be performed at over 1000 mm/hour. Therefore, definition of timeline is impossible without prior experiment definition. Similar considerations hold for all other processes although ranges of solidification velocity may not be so severe.

Equipment selection is also subject to experiment definition. For example, we have selected a platinum furnace as a piece of standard equipment and feel that an 18 inch, 8 shunt unit is a representative item for performing most experiments. However, fewer shunts represent lower power consumption as does smaller volume or length. Possible tradeoffs can be examined only when specific requirements are identified. For the same reason, experiment assemblies to be placed within the equipment has not been defined. It is clear that within the characteristics of the furnaces selected one can get satisfactory volumes of isothermal and gradient regions while sufficient flexibility is available for tailoring these regions to meet specific requirements. The estimated sample volumes are, in fact, selected to permit thermal shaping assemblies to be included in the available volume.

We have selected a representative assembly of equipment on which requirements can be based. For example, if 10 kW is delivered to slightly modified equipment and sufficient heat dissipation facility

is available, all equipment can be run simultaneously and substantial experimentation can be performed during a mission. However, equivalent off-the-shelf commercial hardware may require 2-3 times that power to perform the same number of experiments. More significant modifications to equipment may result in substantial conservation of power over our present thinking.

We have made no attempt to designate measurement and control equipment since this has already been looked at in some detail by TRW and further work would be redundant. We have examined some concepts for performing the appropriate functions and considered possible approaches that could be used within the framework of commercial technology.

7. IMPLICATIONS FOR RESEARCH

It seems clear that furnaces, electronics, and manipulation equipment equivalent to that normally available in a laboratory setting can be accommodated on space missions. In the automatic mode a substantial amount of ground-based data is a requirement to perform an experiment. This is particularly true in the case of those experiments in which manipulation of the sample is required. Some examples of the type of detailed information required would indicate the data requirements.

On frequent occasions it is necessary to obtain a homogeneous melt before solidification occurs. The time for accomplishing this can be determined readily in the laboratory by performing a series of experiments. However, the time in free fall can vary significantly from laboratory conditions in that convective turbulence may not be present and homogenization rates will be limited by such factors as homogeneity of the initial solid, diffusion rates, and particle size. In fact, with moderate sized samples it may be necessary to assist mixing by electromagnetic or mechanical stirring.

Another consideration involves the degree of function that is actually required; for example, cooling or quench rate. It is reasonably straightforward to quench a laboratory sample by simple immersion into a cooling fluid. If it is necessary to perform this quenching in space, a significant technological effort will be required. It becomes desirable then to define what cool rates are truly essential for successful performance and utilize techniques that will meet the objectives within the framework of reasonable facilities -- perhaps gas cooling of fluid cooled molds.

One must also make a detailed evaluation of the chemical systems involved. From Skylab E-beam melting experiments it became

clear that a knowledge of the solid-liquid and vapor phase relations are necessary, in that there is no driving force for vapor to escape from a molten mass. As a result, vapor entrapment (if gas is present) during solidification is a likely event. Such vapor pockets can have a profound influence on the results of experiments. In a system utilizing multiple valence oxides, for example, oxygen is an important part of the system. Consider iron or titanium in glasses. If FeO is the resultant valence state in a material starting with Fe_2O_3, then oxygen pockets can form on solidification causing voids and strains. Similar effects will be observed during crystal growth. As a result, detailed knowledge of the system is a requirement to provide the appropriate starting materials and ambients for successful experiment performance. This factor must be a consideration in any transition metal-chalcogenide system.

A final example of research implications would be a careful definition of what manipulation requirements exist. With encapsulated experiments, the procedures normally used in the laboratory are satisfactory. Unconstrained material processing must be thoughtfully examined. Since "pouring off" is not a viable space process, manipulations such as casting into a mold, quenching, and solvent removal pose interesting problems. Some approaches have been discussed earlier.

8. RECOMMENDATIONS FOR FURTHER STUDY

Further study should include at least two aspects. These are a better definition of potential materials to be used in space experimentation and a description of equipment based on materials processing parameters.

8.1 Materials Experiments

The present plan for the formation of a working group to act as an advisory group on research and processes for materials should be implemented. Among the responsibilities of this committee should be to review the results of space processing experiments to date, examine proposed areas of experimentation and project and recommend experiments to be performed during early missions. As much as possible, the objectives of these experiments should be delineated and the critical parameters in the performance of the experiment identified.

On the basis of these recommendations and projections, a detailed analysis of specific chemical systems should be performed to identify gaps in, for example, knowledge of solid-liquid-vapor phase relations, solidification and/or crystallization behavior, kinetic and thermodynamic processes, and specific processes critical to experiment performance. These should be communicated to the team concerned with equipment selection and design.

8.2 Equipment Selection

On the basis of specific inputs from the materials committee, a detailed equipment analysis can be performed. This should include several aspects.

1. Furnace selections.
2. Flight worthiness and structural analysis.
3. Detailed thermal analysis of furnace.
4. Potential modifications and detailed analysis of modified furnace.
5. Interface requirements (cooling, power, atmosphere).
6. Compatibility analysis of experiments and furnace.
7. Electronics selection.
8. Detailed analysis of function and EMI-RFI compatibility.
9. Potential modifications to components and function analyzed.
10. Interface requirements with furnace and spacecraft.
11. Compatibility of electronics and furnace with materials experiment goals.
12. Analysis of accessory equipment required to perform experiments.
13. Compatibility with automated techniques.
14. Overall analysis of the experiment, furnace, electronics accessory equipment, automation equipment, and spacecraft facility in order that a workable conceptual design of a total experiment package may be developed.
15. Evaluation of the versatility of the concept and its applicability to experiments of the same type and other types.